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# A Compilation of Historical Data on Hagan Drop Testing with Results

Tim Stone, Paul Smith, Tristan Karns

## Background

The Hagan nuclear material storage container was designed in the late 1990's to provide a robust container for daily use, safe transport within the plutonium facility PF-4, and storage of nuclear material for up to twenty years. There are currently >3000 loaded Hagan containers in use at TA-55. The majority of these are in the TA-55 vault, but they are also used in safes and in floor locations on the main floor of PF-4. The original design criteria are given below.<sup>1</sup>

## Original Design Criteria (~1999)

1. Set of five containers ranging in nominal size from one to twelve quart capacities.
2. Each container shall nest into the next larger size, and have a clearance of about two inches on the height and about ½ inch on the diameter.
3. Containers to be 304 stainless steel and have a body wall thickness on 0.020-0.032 inch to allow transmission of radiation for nondestructive assay.
4. Container body shall be seamless, have a flat bottom with rounded inner edges, and a 32 standard finish (easy to wipe clean to allow decontamination).
5. The lids shall be of 304 stainless steel and have a positive closure mechanism such as a clamp or screw.
6. The lid must contain a nuclear materials filter similar to the Nuclear Filter Technology Inc. model NUCFIL-030.
7. The assembled containers loaded to ½ volume with dry sand must withstand a nine-foot drop impacting on concrete (flat on bottom, inverted, side of body, and 45 ° on lid). The container must not rupture or leak. Deformation of the container is expected. A test will be performed by pressurization through the filter hole to 2 psi with the container submerged in water. Container will pass if no bubbles are observed.

The family of Hagan containers including 1Qt, 3Qt, 5Qt, 8Qt and 12Qt sizes is presented in Figure 1.



Figure 1. A complete set of Hagan Containers (latest design).

Confirmatory drop tests were conducted at various times with varying acceptance criteria as a vulnerability assessment of Hagan containers under postulated drop conditions beyond the initial design qualification testing to release for production. The impetus for additional testing was driven by new anticipated storage container performance criteria- now as specified in Manual 44.4.1-1. This testing was performed to understand under what conditions (height, drop orientation, gross weight, container size, lid, design, etc.) Hagan containers can be credited to protect facility workers. Conditions that cause unacceptable loss of containment include container performance issues such as a slipped thread, lid coming off, container cracking, failure of the body to collar weld etc. In the case where the container remains intact different test methodologies were employed to assess degree (as dropped containers were shown to fail higher sensitivity helium leak testing) of release such as bubble count over a specified time interval or collection of a surrogate powder post drop as a particulate release gram amount.

The design of the Hagan container evolved over time, and there is a specific design variable that merits explanation. The early Hagan design had approximately four full threads on the lid. The design was modified to reduce the number of threads to approximately two at some point in time to reduce the chatter during lid closure. After recognition that the “2-threaded” lids might be more vulnerable in a drop, the lid was changed back to the “full thread” design

Hagan tare weights are as follows:

Hagan Size	Hagan Tare WT. (Kg./lbs.)
1-Qt	1.1/2.43
3-Qt	1.8/3.97
5-Qt	2.3/5.07
8-Qt	3.0/6.61
12-Qt	4.3/9.48

## Synopsis of Tests at Various Locations

Drop tests were conducted at NFT in 1998 from four and eight feet onto an unyielding surface in five orientations; on the handle, bottom, side and 45° top and bottom. The package configuration involved loading 3.6 Kg dry sand into a pewter can, PVC filtered bag then a polyethylene filtered bag. The acceptance criteria was based on bubble leak testing under ~ 5" water column internal pressure then submerged in water to check for leakage; considered a pass if no bubbles are detected over a 3 minutes observation interval.

Independent drop testing occurred at LANL (ESA-MT)<sup>2</sup> site in 1999 to determine Hagan design vulnerabilities. Helium leak testing was performed before and after as a quantitative pass/fail with a pass at of  $1 \times 10^{-5}$  or better. It is apparent leak testing was done in an outside in mode pulling a vacuum on the Hagan connected to a mass spec. then spraying helium around the outside to identify any leakage. Masking a leak may occur if the lid remains in place enough post drop that when pulled down onto the body under a vacuum compression on the O-ring is realized in cases that may have otherwise failed the leak test. Units tested included one, three, eight and twelve court sizes, initial drop height onto an unyielding target at 8'; reduced to 6' if failure occurred at 8'. Started with twelve quart as the bounding case and tested up to any of 7 impact orientations.

Drops at NFT Nov./Dec. 2005<sup>3</sup> involved an assessment of particulate release with 20g Magnetite powder as the surrogate material. Payload weights were based on maximum weights stored in the various size Hagans at that time. Test orientation was primarily focused on Center of Gravity over corner (CGOC) onto the lid with additional testing involving side drop onto a bar. It should be noted impact in this side orientation impacting on a bar in the vicinity of the weld always resulted in failure of the weld, creating a large gap. Acceptance criteria was not hard set, primary intent was observation of particulate release from drop heights of 4' and 13' 3"; clearly if the lid came off, or a thread was jumped, or a gap in the weld occurred this was considered a failure. Package configuration involved a bagged stainless steel slip-lid taped containing tungsten shot, surrogate material ~20g Magnetite powder directly loaded in the Hagan.

Drop tests at NTRC<sup>4</sup> involved testing of Hagan containers as a subset of On-site Storage Containers to DOE Draft Manual 441.1-1 Criteria, Nov. 2009<sup>5</sup>. The package configuration was 15 Kg of tungsten shot loaded in a taped slip lid then placed in the Hagan container. Only the 8 Qt Hagan was evaluated from 4', 8' and 12' in CGOC on the lid or side drop orientation. The acceptance criteria involved pre and post drop bubble leak testing at ~ 5 kPa internal pressure and a 30s interval. Bubble leak was not applied post 12' drop as the intent was simply to assess the potential for the lid coming off or jumping a thread at that height. Separate testing was done to assess the ability of the Hagan closure design to pass a helium leak test to a Manual driven design release rate of  $1.3 \times 10^{-6}$  atm cm<sup>3</sup>/s with heat source plutonium as the bounding case to acquire this leakage rate. Hagan testing occurred in the inside out leak test mode at a small differential pressure. It was concluded for the Hagan closure design, it was not demonstrated that it could be consistently assembled and subsequently pass the leak test criteria.

### Historical Hagan Drop Test Review

# Tested	Hagan Size	Gross Weight Kg/lbs.	# Lid Threads (2 or 4)	Drop Orientation Unyielding Surface	Test Height	Acceptance Basis	Results
Testing and Validation of Threaded Lid Vented Nuclear Materials Containers, NFT site, 1998 Package Configuration: 3.6 kg dry sand, pewter can, PVC filtered bag then Polyethylene filtered bag Acceptance Criteria: No bubbles over 3 min. when pressurized to 5" water column							
1	3 Qt	~4.6/10	4	bottom	4 ft.	Water bubble leak	Pass
1	"	~4.6/10	4	CGOC-B	4 ft.	"	Pass
1	"	~4.6/10	4	Top onto Stiff handle	4 ft.	"	Pass
1	"	~4.6/10	4	CGOC-L	4 ft.	"	Pass
1	"	~4.6/10	4	Side	4 ft.	"	Pass
1	"	~4.6/10		CGOC-B	8 ft.	"	Pass
1	"	~4.6/10		Top onto Stiff handle	8 ft.	"	Pass
1	"	~4.6/10		CGOC-L	8 ft.	"	Pass
Independent testing at LANL site (ESA-MT) in 1999 Package Configuration: Internal configuration not specified Acceptance Criteria: Helium leak test with an after drop leak rate of 1x10-5 or better							
1	12 Qt	11/24	4	bottom	8 ft.	Helium Leak	Pass 1x10-6
1	12 Qt	11/24	4	handle	8 ft.	"	Fail
1	8 Qt	7/15.4	4	handle	8 ft.	"	Pass 1.6x10-6
1	12 Qt	11/24	4	Parallel flange sheer	8 ft.	"	Pass 1x10-6
1	12 Qt	11/24	4	handle	8 ft.	"	Pass 1x10-6
1	12 Qt	11/24	4	Parallel flange sheer	8 ft.	"	Pass 1x10-6
1	8 Qt	7/15.4	4	Parallel flange sheer	8 ft.	"	Pass 1x10-6
1	8 Qt	7/15.4	4	bottom	8 ft.	"	Pass 1x10-6
1	12 Qt	11/24	4	Base of lid threads top	8 ft.	"	Repeat 1x10-6
1	12 Qt	11/24	4	Base of lid threads bottom	8 ft.	"	Fail
1	12 Qt	11/24	4	CGOC-L	8 ft.	"	Pass 1x10-6
1	12 Qt	11/24	4	90 deg. Side on body	8 ft.	"	Fail 1x10-2
1	12 Qt	11/24	4	90 deg. Side flange	8 ft.	"	Fail
1	8 Qt	7/15.4	4	CGOC-L	8 ft.	"	Pass 5x10-7
1	8 Qt	7/15.4	4	Base of lid threads 15 deg. On lid	8 ft.	"	2x10-8
1	1 Qt	2/4.4	4	90 deg. Side on body	8 ft.	"	Fail
1	8 Qt	7/15.4	4	90 deg. Side flange	8 ft.	"	Fail

# Tested	Hagan Size	Gross Weight Kg/lbs.	# Lid Threads (2 or 4)	Drop Orientation Unyielding Surface	Test Height	Acceptance Basis	Results
1	3 Qt	3/6.6	4	90 deg. Side on body	8 ft.	“	Fail
1	8 Qt	7/15.4	4	90 deg. Side on body	8 ft.	“	Fail
1	3 Qt	3/6.6	4	90 deg. Side flange	8 ft.	“	Pass 5x10-8
1	8 Qt	7/15.4	4	CGOC-L	8 ft.	“	Pass 5x10-8
1	12 Qt	11/24	4	90 deg. Side on body	6 ft.	“	Fail
1	8 Qt	7/15.4	4	90 deg. Side on body	6 ft.	“	Fail
1	3 Qt	3/6.6	4	90 deg. Side on body	6 ft.	“	Fail
1	1 Qt	2/4.4	4	90 deg. Side on body	6 ft.	“	Pass 1x10-7
1	12 Qt	11/24	4	90 deg. Side on body	8 ft.	“	Fail
1	8 Qt	7/15.4	4	90 deg. Side on body	8 ft.	“	Fail
1	5 Qt	4.5/	4	90 deg. Side on body	8 ft.	“	Fail
1	3 Qt	3/6.6	4	90 deg. Side on body	8 ft.	“	Pass 4x10-8
<p>Drop Tests Results for the Threaded-Top Standard Nuclear Material Container in Nov. &amp; Dec. 2005, Site NFT</p> <p>Package Configuration: Bagged stainless steel slip-lid taped containing tungsten shot, surrogate material ~20g Magnetite powder directly in Hagan</p> <p>Acceptance Criteria: Failure a jumped thread or lid removal, otherwise assess surrogate release</p>							
10	1 Qt	3/6.5	2	CGOC-L	13 ft. 3”	Evaluate Release	10- Lid Intact, visual release
6	3 Qt	8.1/18	2	CGOC-L	13 ft. 3”	“	1-Lid came off, 1-JT, 4- Lid intact, visible release
6	5 Qt	1.2/26	2	CGOC-L	13 ft. 3”	“	5-Lid Intact, 1 with significantly more powder
6	8 Qt	1.6/34.5	2	CGOC-L	13 ft. 3”	“	4- Lid Intact, 1with damaged threads difficult to open, 1 unscrewed lid by 1 ¼” and lid could not be removed, visible release
6	12 Qt	20/44	2	CGOC-L	13 ft. 3”	“	1-Lid came off, 5- Lid Intact
4	1 Qt	3/6.5	2	Side or side handle & bar	13 ft. 3”	“	2-failed weld, 2 intact with visible release
3	3 Qt	8.1/18	2	Side or side handle & bar	13 ft. 3”	“	2- failed weld, 1-Intact with visible release
3	5 Qt	1.2/26	2	Side or side handle & bar	13 ft. 3”	“	2- Intact
3	8 Qt	1.6/34.5	2	Side or side handle & bar	13 ft. 3”	“	1 failed weld, 2- Intact one of which visible release
4	12 Qt	20/44	2	Side or side handle & bar	13 ft. 3”	“	1 failed weld, 3 Intact two of which visible release
1	1 Qt	3/6.5	2	Bottom	13 ft. 3”	“	Intact
1	12 Qt	20/44	2	Bottom	13 ft. 3”	“	Intact
1	1 Qt	3/6.5	2	CGOC-L	4 ft.	“	Lid Intact

# Tested	Hagan Size	Gross Weight Kg/lbs.	# Lid Threads (2 or 4)	Drop Orientation Unyielding Surface	Test Height	Acceptance Basis	Results
1	3 Qt	8.1/18	2	CGOC-L	4 ft.	“	Lid Intact
1	8 Qt	1.6/34.5	2	CGOC-L	4 ft.	“	Lid Intact
1	12 Qt	20/44	2	CGOC-L	4 ft.	“	Lid Intact, visible release
2	1 Qt	3/6.5	2	Side or side handle & bar	4 ft.	“	2- Intact
2	3 Qt	8.1/18	2	Side or side handle & bar	4 ft.	“	2- Intact
2	8 Qt	1.6/34.5	2	Side or side handle & bar	4 ft.	“	2- Intact, both visible release
2	12 Qt	20/44	2	Side or side handle & bar	4 ft.	“	2- Intact
1	1 Qt	3/6.5	2	bottom	4 ft.	“	Intact
1	12 Qt	20/44	2	bottom	4 ft.	“	Intact
3	1 Qt	3/6.5	4	CGOC-L	13 ft. 3”	“	3- Lid Intact with all visible release
3	3 Qt	8.1/18	4	CGOC-L	13 ft. 3”	“	3- Lid Intact with 2 visible release
3	5 Qt	1.2/26	4	CGOC-L	13 ft. 3”	“	3 Lid Intact with 2 visible release
3	8 Qt	1.6/34.5	4	CGOC-L	13 ft. 3”	“	3- Lid Intact with 2 visible release
3	12 Qt	20/44	4	CGOC-L	13 ft. 3”	“	2- Lid Intact with one visible release, 1-JT
NTRC Testing of Hagan containers as a subset of On-site Storage Containers to DOE Draft Manual 441.1-1 Criteria, Nov. 2009 Package Configuration: Tungsten shot in taped slip lid cans Acceptance Criteria: Helium leak tested empty, then pre and post bubble leak testing after loading over 30s at ~5 kPa internal pressure							
2	8 Qt	15/33	4	CGOC-L	8 ft.	Water bubble leak test	1-JT, 2-Fail-bubbles too numerous to count
1	8 Qt	15/33	4	CGOC-L	4 ft.	“	1-Fail-bubbles too numerous to count
1	8 Qt	15/33	4	CGOC-L	12 ft.	Bubble test not done	1-JT 180° from POI- Fail
1	8 Qt	15/33	4	Intended CGOC-L (was slap down)	12 ft.	“	1-Fail significant bubble count
1	8 Qt	15/33	4	Side on handle	8 ft.	Water bubble leak	1-Fail- fewer bubble count
1	8 Qt	15/33	2	CGOC-L	8 ft.	“	1-Fail- bubbles too numerous to count
1	8 Qt	15/33	2	CGOC-L	4 ft.	“	1-Fail- bubbles too numerous to count
Totals?							



## Summary

Testing and Validation of Threaded Lid Vented Nuclear Materials Containers, NFT site, 1998:

NOTE: Results expressed here were based on initial design qualification evaluation.

The results of the air-leak after a 1.21 meter (4 ft.) drop test indicates that the container seal was not compromised from the drop impact. Hydrogen diffusivity measurements indicate hydrogen gas will be transported through the integral filter and semipermeable membrane at a rate of about  $15 \text{ E-06}$  moles/second/mole fraction. Filter efficiency tests demonstrate that a reliable seal is formed with the sintered stainless steel filter media. Particle retention of 0.3 to 0.5 micron DOP aerosol was measured at greater than 99.97% at an air flow of greater than 210 milliliters per minute. Through three different water entry tests, one pressurized at 1 PSIG and two water spray tests, it is demonstrated that water will not enter the container.

Independent testing at LANL site (ESA-MT) in 1999:

Note: Initial design qualification testing, the single inside weld design option is what went forward in manufacturing of Hagan containers as a point of clarification to the synopsis below.

Most of the 90° side of body orientation drops breached the weld next to the threads, pulling them away from the lid and breaking the seal between the lid and body of the can. The drop test failures in this orientation caused us to strengthen the area below the lid to maintain a leak free seal. After consultation, a collar was added to the one, three, and eight quart containers and lengthened on the twelve-quart container. The collar was welded inside at the top and outside on the bottom (new generation containers). A new five-quart container was being added to our inventory at LANL so it was fabricated with the double weld collar (NMC2-05-1). The new generation containers with double weld collars were then dropped in the 90° side of body orientation as before (NMC2-012-2, NMC2-08-2, NMC2-05-1 and NMC2-03-1). The new generation three-quart container passed the drop test at eight feet; the larger containers failed. The new generation container proved to be superior to the original container. The cost of the new generation container will be substantial due to the extra fabrication and welding. LANL is

evaluating cost versus gain and will make a decision to convert to the new container or continue to use the existing container in the future. Whether we use the original nuclear material container or the new generation container, we have a safer, easy to use container with many innovative features.

Drop Tests Results for the Threaded-Top Standard Nuclear Material Container in Nov. & Dec. 2005, Site NFT:

Note: One noteworthy practice in implementation of the SNMC/Hagan storage container design was the implementation of a standard packing configuration; which for residues and oxides involved a taped slip lid then a bag out bag which was then placed in the Hagan. Testing as indicated in this synopsis does not credit these inner layers since the surrogate Magnetite powder was purposefully placed directly inside the Hagan prior to testing.

From the data collected, there is a vulnerability regarding the SNMC during a side impact where the container strikes a surface such as a steel bar in the vulnerable area. To recap, the vulnerable area is between the top of the side-handle to the lid, 360 degrees around the SNMC.

We see that the SNMC has some serious tendencies in the event of the abnormal condition, a drop. This suggests careful handling practices for the SNMC that contains dispersible radioactive or nuclear material. We do recognize that in order for a puff to occur, the inner container and bag must fail, and dispersible material must be directly inside the SNMC, so that in the event of a drop, there is high likelihood that a certain amount of that dispersible material will be puffed SNMCs.

NTRC Testing of Hagan containers as a subset of On-site Storage Containers to DOE Draft Manual 441.1-1 Criteria, Nov. 2009:

Note: When the synopsis below refers to the Design Qualification Release Rate this is a greatly relaxed release rate allowed per Manual M441.1-1 as a post drop release rate criteria and is on the order of  $10^{-3}$  cc/s as compared to a much more stringent Design Release Rate of more like  $10^{-6}$  cc/s as an equivalent leakage value based on plutonium materials in storage. In any case, if the container being tested can pass the Design Qualification Release Rate it would be acceptable per the Manual.

In general the test units fared poorly when compared to the criteria contained in the Draft DOE Manual 441.1-1. The following observations are provided:

- For the design release rate tests, two SNMCs/Hagans were tested for comparison to design release rate criteria, and one of these units was tested several times. Although TU-1, Test 1, retest 2, TU-1 Test 2, retest 1, TU-1, Test3, retest 1 and TU-5 passed this test, it was not demonstrated that the SNMC design could consistently be assembled and subsequently pass the design release rate criteria. Since containers used in the field

cannot be leak tested when in use, it is important that it can be shown that use of a standard assembly procedure consistently results in a high-quality (i.e., leak tight) seal.

- For the Design Qualification Release Rate, it was found that the SNMC designs, after drop testing from either 8 feet or 4 feet could not meet the Design Qualification Release Rate criteria.

## REFERENCES

1. Gladson, J.; Hagan, R. *Nuclear material container testing*; LA-UR-99-3709; Los Alamos National Laboratory: Los Alamos, NM, 1999.
2. Terry Wickland; Marty Mataya, Testing and Validation of Threaded Lid Vented Nuclear Materials Storage Containers
3. Cindy J. Mills et.al., Drop Test Results for the Threaded-Top Standard Nuclear Material Container
4. M. R. Feldman; James O'Brien, Test Report on Testing of Los Alamos National Laboratory On-Site Storage Containers to DOE Draft Manual 441.1-1 Criteria
5. James O'Brien, DOE M 441.1-1 Chg 1 (Admin Chg), Nuclear Material Packaging

## Attachment A

# **Drop Test Results for the Threaded-Top Standard Nuclear Material Container**

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Timothy A. Stone, PMT-3, December 2007  
Tresa F. Yarbrow, PMT-3, December 2007

## **Abstract**

Nuclear material handlers use the Standard Nuclear Material Containers (SNMCs) for temporary storage of nuclear and radioactive materials. These SNMCs are found stored in the Los Alamos National Laboratory (LANL), Technical Area 55 (TA-55), Plutonium Facility (PF-4) nuclear materials storage vault. In the nuclear materials storage vault, SNMCs are stored as high as 9.5 feet. Program Managers decided that LANL personnel should arrange for drop tests, to determine how well the SNMC would perform during a drop test. Later, it was determined to drop the SNMC from 1.2 times the maximum storage height, which was the multiplication value documented in the draft DOE Packaging Manual.

Nuclear Materials Technology Group, NMT-4 personnel arranged for conducting drop tests at Nuclear Filter Technology (NFT) for the five SNMC sizes. (Nuclear Materials Technology is currently Plutonium Manufacturing Technology, and Group NMT-4 is currently PMT-3.) In September, November and December, NFT conducted drop tests of SNMC, with NMT-4 personnel observing the tests.

Initially, in the September time frame, the thought was to drop SNMCs and after each test, subject the SNMCs to a Bubble Leak Test, per NMT-4's Systems Engineer. Upon performing the leak test, the SNMCs failed miserably. These drop tests were not thoroughly documented.

In November, NFT performed additional drop tests employing two additional criteria, per the Systems Engineer. These criteria were using a surrogate material directly inside the SNMC and dropping the SNMCs in the orientation of center of gravity over center (c-g/c).. We used a surrogate material to mimic the respirable fractions of plutonium oxide, and we mimicked the packaging of a worst-case scenario. A typical packaging configuration includes an inner stainless steel slip-lid container, in which the material is put. This inner container is taped closed using two wraps of vinyl tape. The inner container is then placed inside a thick walled plastic bag, which is also taped closed with vinyl tape. This bagged inner container is then placed into an SNMC. In all packaging issues, it is rare that material gets out of the inner container and the bag; however, we put the surrogate material directly inside the SNMC, to mimic a failure of the inner container and bag. These tests were documented thoroughly.

## Abstract, continued

Because these drop tests were documented so thoroughly, the data yielded two distinct sets of results. The first set of results yielded the following: Two of the drops resulted in the complete removal of the lids. The two SNMC sizes that lost lids were the 3-Quart and the 12-Quart. Losing the lid resulted in a complete breach or loss of surrogate material. Of particular note, these lids were of the 2-threads design. The second set of results yielded the following: The remaining drops resulted in small amounts of surrogate material outside each SNMC, which indicated a small puff of surrogate material exited the SNMC, upon impact.

After reviewing the November test results, the Program Managers wanted additional criteria added to the drop tests to be conducted in December. These criteria were having full- or 4- thread lids manufactured, and to capture the released surrogate material.

In December, Full- or 4-thread lids were used on SNMCs, and this array dropped. No Full- or 4-thread lid came off. Of interesting note is that no 2-thread lid came off. NPT personnel were also able to recover the surrogate material puff, using a technique suggested by the LANL NMT-4 representative. In addition, other orientations (side and bottom impact sites) were used, and a different drop height of 4 feet.

With regard to the side-orientation impacts, SNMCs struck a 1" steel digging bar, to mimic an impact onto a transfer vehicle, used for transporting material in PF-4. We found that side-orientation onto a bar impact yielded more a vulnerable impact orientation than the cg/c orientation drops. On the SNMC, there is a circumferential area in the region of the bottom of the lid to the side-handle, where the SNMC is vulnerable to denting and subsequent moving of the body away from the lid, resulting in a breach or complete loss of surrogate material.

Regardless of the height of the drop, drop orientation, or whether the SNMC was fitted with a 2-thread or 4-thread lid, a surrogate material puff at impact was always emitted. Using correction factors, the range in puff size was between 1 to 10 mg.

After performing drop tests of the Threaded-Top Standard Nuclear Material Container (SNMC), and reviewing the data recorded from the three different months of drop tests, we know there are limitations of the SNMC, during an upset condition; specifically, a drop test.. The concern about this vulnerability is if dispersible material such as plutonium oxide somehow breaches the taped inner container and the taped bag, the SNMC would not contain or protect a material handler, in the event of the abnormal condition, a drop.

## Definitions

Word or Phrase	Definition
2-thread SNMC	There are two threads machined into the inside of the vertical surface of the lid.
4-thread SNMC	There are four or full threads machined into the inside of the vertical surface of the lid.
breach	Upon impact, a lid came off, a weld tore, or the body threads were deformed away from the lid threads.
catastrophic	A resulting situation where a breach causes a total loss of powder containment.
extension collar	A collar that is welded onto the Vollrath or Polarware stainless steel container to make the taller 12-quarts container. The thread-ring collar is then welded to the extension collar.
non-catastrophic	A resulting situation where there was a partial loss of containment.
powder puff	An observed phenomenon for all non-catastrophic drops, where an amount of powder exits the SNMC (exclude the threads) and is deposited on the outside of the SNMC (exclude the material in the thread) or in the collection bag.
side-drop vulnerable area	An area from the side-handle to the bottom of the lid, 360 degrees around that is vulnerable during a side-drop onto a stainless steel bar. If an impact occurs within this area, there is a high probability that a catastrophic event will occur.
side-handle	Refers to the vertical flange welded to the side of the SNMC, in two places. A hole must be located in the top of the side-handle, for accommodating a Tamper Indicating Device (TID) wire for applying a TID. The side-handle is also used to hold a pewter shield overpack to the SNMC.
thread-ring collar	A collar welded to the top of a Medegan or Polarware deep drawn stainless steel container. Once welded in place, external threads are cut into the collar, creating the external body threads.

## Introduction

Los Alamos National Laboratory (LANL) Technical Area 55 (TA-55), the Plutonium Facility (PF-4) supports the use of Threaded-Top Standard Nuclear Material Containers (threaded-top SNMC or known within the TA-55 Facility, as the Hagan Container), for interim packaging and storing of radioactive and nuclear materials. The inception of the SNMC begins as an assignment given to Roland C. Hagan, a LANL Technical Staff Member (retired), by Mr. Hagan's Group Leader, Dennis L. Brandt (retired). This assignment came about as the result of the Defense Nuclear Facility Safety Board (DNFSB) Directive 94-1, a vault container inspections effort [1]. Mr. Hagan worked to establish container inspection criteria, for inspecting vault containers. From completing the inspections and reviewing data, Mr. Hagan identified the most common storage container issues. His container design idea took into consideration user safety and various attributes, including but not limited to having few parts, a positive closure lid, no sharp edges, easy to use, vented, and capability for use with existing nondestructive assay techniques. Mr. Hagan suggested a design idea for standard containers, which he and Mr. Brandt recommended to the TA-55 management team, comprised of group leaders and alternate group leaders. The management team consensus was that the container idea had merit. After the management team authorized the design, Mr. Hagan wrote his requirements in a memo, and these requirements went out in a LANL Solicitation. Five companies responded to the Solicitation, with two of the five manufacturing container prototypes, for evaluation. Mr. Hagan received prototypes from these two companies, and after careful consideration, he chose Nuclear Filter Technology, Inc. (NFT) to manufacture his container design idea. Figure 1 shows the five different sizes of SNMCs, and it identifies a few of the parts and areas, of SNMCs, discussed in this document.

## Introduction, continued



Figure 1

## Introduction, continued

**Figure 2** shows two container bodies with the exterior threads, lids, and pewter shields.



**Figure 2**

Mr. Hagan and NFT, conducted drop tests from approximately 4' and 8' heights. To establish a ground point, the SNMCs were bubble leak tested before the 4' or 8' drop. Passing criterion was for the SNMC to pass a bubble leak test, after the drop. [2], to verify a sealed condition, after the drop impact.

The LANL Materials Engineer conducted the second set of drop tests in January, March, and June 1999. The LANL Materials Engineer performed 8' drop tests of 4-thread 1-quart (Q), 3Q, 8Q, and 12Q SNMCs. There were different orientations or point of impact sites. Included among the different orientations were side-type drop orientations, including side impacts and side impacts on the side-handle. Out of fifteen side-type orientation drops, there were twelve failures, and the three passing SNMCs may have suspect leak tests. The drop test results indicated the need for a more robust thread-ring

## Introduction, continued

collar, and other enhancements [3]. NFT manufactured enhanced prototypes, which were also drop tested. The three largest sizes, the newly introduced 5Q, the 8Q and the 12Q, even with their collar enhancements, each failed the 8' drop test. The 1Q and 3Q did pass the 8' drop test. The enhanced SNMC would cost more money to manufacture, and a cost versus gain evaluation was to occur. (There are no written records regarding ever doing this assessment. By hypothesis, somebody evidently made a decision to not proceed with the enhanced model.) In the mean time, the existing design was in use for storing radioactive / nuclear materials.

## Drop Tests

The August 5, 2003 plutonium-238 uptake incident, resulted in a number of findings. One finding in particular, required LANL group NMT-4 to complete packaging requirements document. There were a number of design requirement tests implemented in this requirements document NMT-RD-003 [4]. One requirement was to drop the SNMC from the highest storage height of the Chemistry Metallurgy Research Building or TA-55 storage vault. Another requirement was to helium leak test the container using the Envelope-type method to the "Leak Tight" value stated in ANSI 14.5, *American National Standard for Radioactive Materials – Leakage Tests on Packages for Shipment*. The other motivation for the November and December 2005 drop tests was the draft Department of Energy (DOE) Packaging Manual, and TA-55 chose to be proactive about ascertaining how the 2-thread SNMC responded in a worst-case upset condition. In this case, the worst-case condition is where we simulated plutonium powder loaded directly in contact with the SNMC, the outside container. To achieve this condition, we simulated failures of the standard inner package, an approved inner container and an approved bag. Another part of the test was to drop the SNMC from 1.2 times the highest storage location in the TA-55 nuclear materials storage vault. This 1.2 times factor was the value given in the DOE Packaging Manual. The height of 13'3" was derived from multiplying the factor to height of the top front of a tall Bisco Drawer in Room I. This height value is very conservative, because there is only one location for which this situation would apply. Administrative controls could be implemented to prohibit SNMCs containing oxide from being stored in the two locations that might cause an operator to rest a container on this particular surface. What this means is that a degree of conservatism was built into the drop height, with respect to the drop height required in the draft DOE Packaging Manual, of that time. Of interest is this 13'3" drop height is approximately 1.4 times the highest storage height of the TA-55 nuclear materials vault.

In the past, drop test powder materials used to simulate a worst-case situation, included Fluorescein or chalk. Fluorescein requires the use of a carrier powder, for detection. Typically, personnel use flour as the carrier for Fluorescein. The issue with using chalk or Fluorescein on powder is the particle size. The respective particle sizes for

## Drop Tests, continued

Fluorescein with flour and chalk are respectively 30 to 180 microns and 3 to 30 microns. Respirable plutonium powder has a particle size range of 0 to 3 microns. To simulate respirable plutonium powder, we opted to use Magnetite Powder, having a nominal particle size range of 0 to 5 microns. To further simulate an actual standard package containing nuclear material, in addition to using the Magnetite powder we used additional materials.

To simulate an actual standard package containing nuclear materials, we referred to the authorized NMT-Division Work Instruction Document, at that time, NMT-WI-021, Packaging, Handling, Storing, and Moving Radioactive Materials. NMT-WI-021 mandated the use of Vollrath stainless steel slip-lid containers for the inner containers, approved bags, and approved vinyl tape. To simulate the greatest weights of items currently stored in the TA-55 nuclear material vault, NFT procured small B-B sized tungsten shot and fully loaded the inner stainless steel slip lid container, with tungsten shot. Refer to **Figure 3**, for the tungsten weights of the different sizes of slip-lid containers.

SNMC Size:	1-Quart	3-Quarts	5-Quarts	8-Quarts	12-Quarts
Load Weight (lbs.)	4	14	21	28	35
Slip-lid Container Size (qt.)	$\frac{5}{16}$	2	$2\frac{7}{8}$	$5\frac{1}{2}$	$8\frac{1}{2}$

**Figure 3**

To perform the November 2005 or first set of drop tests, NFT completed helium leak tests and setups of Drop Test Equipment.

## First Set of Drop Tests

In approximately August 2005, we discovered that the 1Q SNMC could not pass the new helium leak test leak rate specified in NMT-RD-003. In September 2005, we discovered that the 3Q SNMC could not hold any of the pressure specified in NMT-RD-003. Before beginning the November 2005 drop tests, NFT completed a mass-spectrometer helium leak test in the Tracer Probe Mode, to verify weld-integrity of all SNMC welds. LANL verified these preliminary leak tests. Before beginning the leak tests, the NFT helium leak tester, Level 2, tightens an adapter into the filter port of each SNMC, and the mass spectrometer pulls a vacuum on each SNMC. The leak tester moves the probe spraying helium across weld locations, while using a standard scan rate. If there were any cracks or holes in a weld, the vacuum “pulled” helium into the mass-spectrometer, and the mass-spectrometer detected the amount of helium. This testing method is good for results measuring at least  $1 \times 10^{-6}$  std-cc/sec. Each SNMC used in the drop tests, passed a leak test. NFT used a Drop Test Fixture, [4] Of note is the fact that the Drop Test Fixture had a manual crank winch versus the electric winch used in December 2005.

NFT personnel loaded each helium leak tested Container with a bagged stainless steel slip-lid container containing tungsten shot. Personnel tightened the lid to the body, where the lid closure mark was 1/4" past the body closure mark. Before dropping each Container, NFT personnel placed a bag around it. NFT personnel placed the bagged SNMC onto the Drop Test Fixture, beginning with the smallest SNMC, a 1Q size, and ending with the largest SNMC, a 12Q size. NFT dropped seventeen 2-Thread SNMCs in the pre-determined vulnerable Center of Gravity/Center (c-g/c) position. The c-g/c position was a point on the edge of the lid, near the filter, which was 45 degrees from the steel drop surface. We have numerous movie files taken with my personal camera to verify each Container impacted the surface, in this c-g/c orientation. What was surprising during these drop tests is that there were two catastrophic events, where the lid popped off from the side opposite the impact area. The catastrophic events occurred to one 3Q and one 12Q SNMCs. Refer to **Figure 4**, for a picture of a catastrophic event.

Catastrophic  
failure



**Figure 4**

## **First Set of Drop Tests, continued**

In addition, personnel observed two other phenomena. These phenomena are as follows:

- For those non-catastrophic events, personnel noted a small amount of powder in the bag, and
- after opening each SNMC, personnel observed an accumulation of powder in the first two body threads. Refer to **Figure 5**, for a picture showing the powder accumulation in the first couple body threads.

These results were discussed, at length at LANL.



**Material collected in first two body threads.**

**Figure 5**

After discussion at LANL about the November 2005 results, the outcome included three decisions to continue performing drop tests to obtain additional information. One decision was to conduct further drop tests, both for 2-thread and 4-thread lids. Of interest was how the 4-thread lids compared to the 2-thread lids. In addition to conducting drops of the 4-thread lids, we needed to collect and weigh the amount of the non-catastrophic puffs. The third decision was to complete these drop tests before the end of the year. After arranging with NFT to complete additional drop tests through LANL Procurement, and Buyer, Dale Carmichael, a schedule for conducting the drops during the week of December 11, 2005, was decided upon, so that we completed drop tests before the end of the year.

## Second Set of Drop Tests

LANL and NFT completed additional drop tests at the NFT Site. NFT manufactured 4-thread lids for use with SNMC bodies supplied by LANL. To recover puff, we tried different methods. We unsuccessfully tried a couple of ideas, and they did not prove to be effective recovery methods, so I will not go into those ideas here and will not include the data. We finally discovered a good recovery method.

The good recovery method involved using a piece of Blue Masking Tape (blue tape), having a specified adhesive quality. To establish the recovery capability of the blue tape, we completed two standards. The two procedures for establishing the recovery capability are as follows:

### First standard

- Verify the scale is level.
- Zero the scale.
- Tare the blue tape.
- Open an empty bag.
- Smear the adhesive side of the blue tape over the inside surface of the bag to determine how much normal airborne particles apply to our results. This powder also has an affinity to gloves. Unfortunately, we could not use gloves for recovering powder, because they will not remain still enough to obtain a weight.
- Weigh the tape. In this case, the tape picked up 0.060 mg.

### Second standard

- Verify the scale is level.
- Zero the scale.
- Tare the blue tape.
- Put a known quantity of powder in a bag. In this case we measured 0.332 mg into the bag.
- Open the bag and smear the adhesive side of the blue tape over the surface of the bag to determine percent pick up of the powder puff. When opening the bag, we probably lose some powder because of the agitation. People moving, etc. Any holes in bags caused by the impact or bouncing on the surface.
- Weigh the tape. In this case the tape picked up 0.137 mg, which is approximately 43.7% of the original quantity of powder weighed into the bag. I apply this 43.7% value as a correction factor to all quantities picked up by blue tape.

### **13'3" Drop Heights**

We dropped eighteen 2-thread SNMCs and fifteen 4-thread SNMCs, from 13'3". The orientation was c-g/c. All drops yielded non-catastrophic events, but the powder puff was evident from every dropped SNMC.

Then, we dropped fifteen 2-thread SNMCs in a side orientation onto an approximately one-inch steel digging bar, from 13'3". Refer to **Figure 6** for a picture of the Drop Test Fixture, a 1Q SNMC that is oriented for a side drop, and the digging bar. We observed two catastrophic events, and for the remaining non-catastrophic events, we observed the puff. For the two catastrophic events, we saw the impact pushes the body threads away from the lid threads, or the weld that holds the collar to the body, breaks. Refer to **Figure 7**, for a picture of the breach of a catastrophic event. We got very good at predicting the strike site where the SNMC was vulnerable to a breach. We opted not to breach every SNMC but to obtain more puff data.

While we were in the midst of completing 1Q SNMC side-drop tests, we exhausted our blue tape supply. Arnold Brassell, NFT Floor Operations Manager, left to procure a roll of blue tape. All that Mr. Brassell found was a roll of white Masking Tape (white tape) having a slightly different adhesive quality compared to the blue tape.. We subjected the white Masking Tape to the same standard procedures as the blue tape. By completing these standards for the white tape, we observed some very interesting results.

These standard results included the following:

- The blank bag smear yielded 0.060 mg. Although this value is the same value obtained using blue tape, it is noted that the blue tape was capable of recovering only 43.7%% of what dust was actually present. Of course there are other factors to consider, such as turbulence in the air caused by personnel walking by, doors closing, heater fan, etc. This turbulence can cause more or less dust or powder to be present for recovery. But, this test indicated the white tape had excellent recovery capability.
- We loaded another bag with 9.22 mg of powder. Of this 9.22 mg load, we recovered 9.97 mg of powder. If we subtract 0.060 mg from 9.97 mg of powder, we obtain a value that is incredibly close to the original load, indicating the white Masking Tape recovers 100% of load. To be on the conservative side, I will use 90% when I correct values obtained from using white tape.

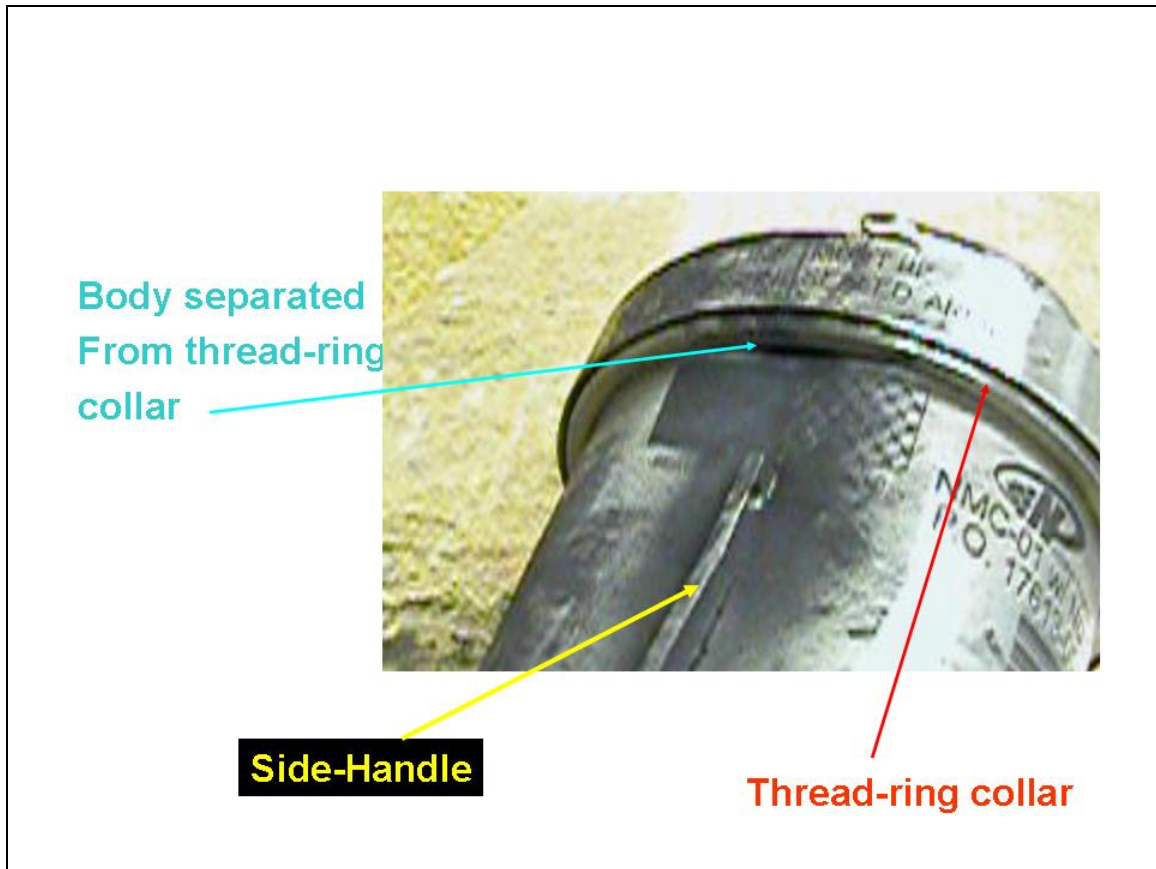
### 13'3" Drop Heights, continued

1-1/2" steel bar



**Figure 6**

## 13'3" Drop Heights, continued



**Figure 7**

Finally, we dropped two SNMCs, at separate times, onto the bottom, from 13'3". The 1-quart and 12-quart SNMCs did not breach, but we did observe the puff.

## 4' Drop Heights

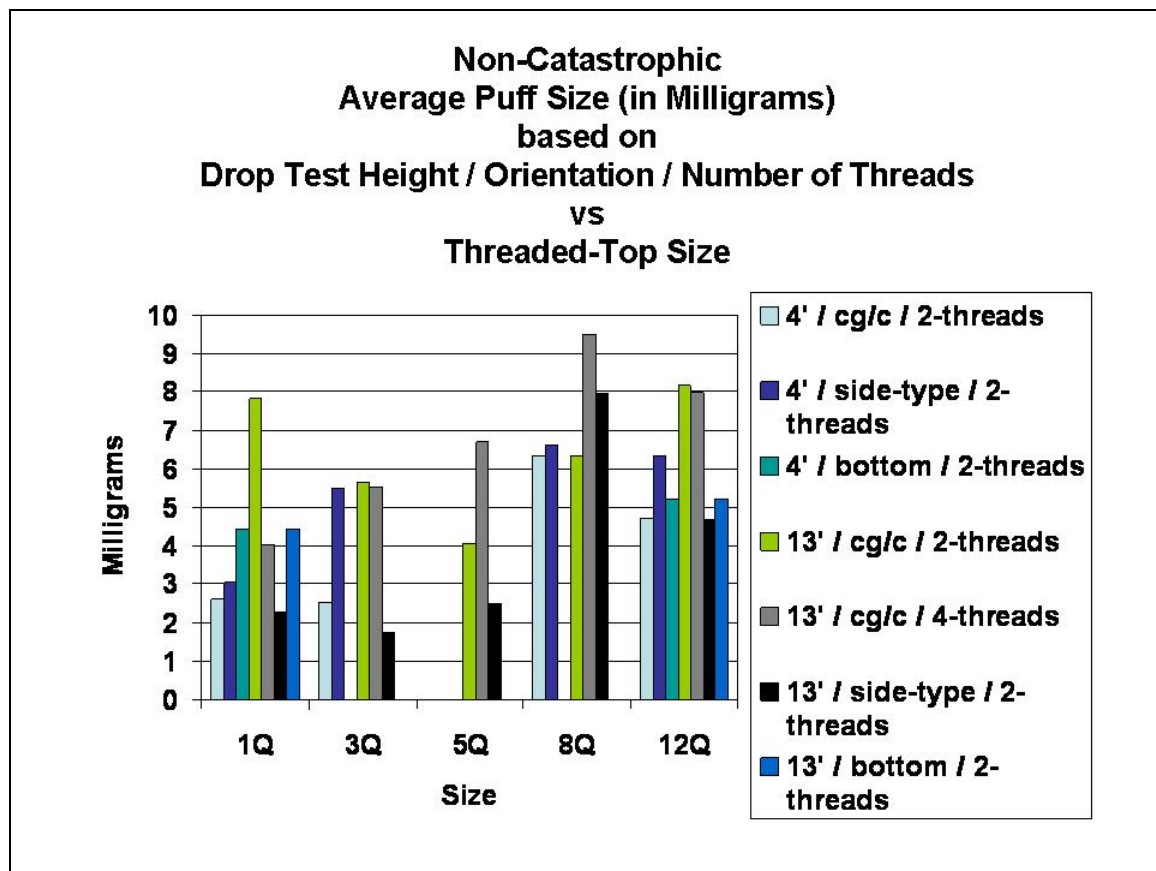
We dropped four 2-thread SNMCs, from 4'. The orientation was c-g/c. All drops yielded non-catastrophic events, but the powder puff was evident from every dropped SNMC.

Then, we dropped eight 2-thread SNMCs onto an approximately one-inch steel digging bar, from 4'. We observed no catastrophic events, but we observed the puff from each SNMC. From the 13'3" side-drops, we knew the vulnerable strike area to cause a breach, but we were not successful in causing a breach in any SNMC during the 4'-drops. This inability to cause a breach was disappointing, but showed the SNMC is relatively safe from a catastrophic failure from this drop height. However, the puff was still evident.

Finally, we dropped two 2-thread SNMCs, at separate times, onto the bottom, from 4'. The 1Q and 12Q SNMCs did not breach, but we did observe the puff.

## All Drop Tests

In some situations, we could not see the puff collected on the inside of the bag, but we could see the accumulation on the blue or white tape. Researching these observations, we found that the human eye can only see particles that are approximately 40 micron or larger, in size. Until these tiny particles are recovered with tape and accumulated in one relatively small area, they were not discernable. Every non-catastrophic drop yielded a puff, regardless of the drop height and the drop orientation. Refer to **Figure 8** for a graph showing the range of an average puff for a particular drop height, orientation, and number of lid threads, versus the size of SNMC. The only values that are not averages are the bottom-type drop results.



**Figure 8**

To further enhance the results obtained from 2005 drop tests, in May 2007, NFT performed four additional tests to prove that powder did not get into the first two body threads, from normal handling. A 1Q and a 12Q each received a bagged loaded inner container and approximately 20 grams of powder. Each lid was threaded to each body, and then, the lid was removed. The body threads were inspected for powder, and no powder was visible. A second set of tests were performed. A 1Q and a 12Q each received a bagged loaded inner container and approximately 20 grams of powder. Each

lid was threaded to each body, the SNMC inverted, the SNMC righted, and then, the lid was removed. The body threads were inspected for powder, and no powder was visible.

## Results

Out of sixty-five drops, there were seven catastrophic failures. Of 13'3" drops, there were two catastrophic c-g/c drops of 2-thread SNMCs, out of a total of 35 drops. This is a failure rate of approximately 5.8%. There were two SNMCs that had 2-thread lids that jumped a thread, a 5Q and a 3Q. There were no catastrophic c-g/c drops of 4-thread SNMCs. There was one 4-thread lid that jumped a thread, a 12Q. There were five catastrophic events from side drops out of a total of sixteen drops for a failure rate of approximately 31%. This drop seems to be the SNMC's vulnerability, even over the anticipated c-g/center drops, although we do not have data to support whether the 4-thread SNMC would also be vulnerable to the side drops. Once we discovered the vulnerable area of impact, we could have breached every side-type dropped container. Instead, we elected to obtain puff data. For every non-catastrophic drop, the SNMC emitted a powder puff. This phenomenon seems to occur during impact and occurs regardless of size of SNMC, drop height, number of lid threads, or orientation. Another phenomenon that seems to occur during impact is the accumulation of powder in the body threads. This phenomenon also seems to occur regardless of size of SNMC, drop height, number of lid threads, or orientation.

Comparing the 2-thread and 4-thread lids during 13'3" c-g/c drops, we see a tendency toward the 2-thread lids coming off, thereby resulting in catastrophic failures.. Although, we see this tendency, we can also make an argument that the 4-thread lid was not dropped as many times as the 2-thread lid. In addition, the 2-thread lid only came off during the November drop tests. There is not enough data for making a hypothesis about why this occurred.

## Conclusions

From the data collected, there is a vulnerability regarding the SNMC during a side impact where the container strikes a surface such as a steel bar in the vulnerable area. To recap, the vulnerable area is between the top of the side-handle to the lid, 360 degrees around the SNMC.

We see that the SNMC has some serious tendencies in the event of the abnormal condition, a drop. This suggests careful handling practices for the SNMC that contains dispersible radioactive or nuclear material. We do recognize that in order for a puff to occur, the inner container and bag must fail, and dispersible material must be directly inside the SNMC, so that in the event of a drop, there is high likelihood that a certain amount of that dispersible material will be puffed. SNMCs

## Recommendations

It would be interesting to see side-drop tests for 4-thread lids being dropped onto a digging bar. In addition, to see the results after dropping 2-thread and 4-thread lids onto an actual transfer vehicle. Also 4-thread lids c-g over c.

SNMC Surveillance should not only test filters and seals, but it should also include radiography and/or mandatory opening/inspecting the inner bag and container, especially for those material matrices stored in SNMCs that may have dispersible tendencies.

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# Appendices

## Appendix A:

November 2005 Drop Test Results	Orientation: Center of Gravity / Center
Lid: 2-thread	Drop Height: 13'3
Object: To strike on edge of lid near filter.	

## Appendix B:

November 2005 Drop Test Results	Orientation: Side / Side-handle
Lid: 2-thread	Drop Height: 13'3
Object: To simulate a drop onto the handles or side of a transfer vehicle.	

## Appendix C:

December 2005 Baseline Results	Baseline run for Blue Masking Tape
Object: To determine the recovery capability of the Blue Masking Tape, and to determine how much of the recovery weight is from dust or pollutants.	

## Appendix D:

December 2005 Drop Test Results	Orientation: Center of Gravity / Center
Lid: 2-thread	Drop Height: 13'3
Object: To strike on edge of lid near filter.	

## Appendix E:

December 2005 Drop Test Results	Orientation: Center of Gravity / Center
Lid: 4-thread	Drop Height: 13'3
Object: To strike on edge of lid near filter.	

## Appendix F:

December 2005 Drop Test Results	Orientation: Side / Side Handle
Lid: 2-thread	Drop Height: 13'3
Object: To simulate a drop onto the handles or side of a transfer vehicle.	

## Appendix G:

December 2005 Drop Test Results	Orientation: Bottom
Lid: 2-thread	Drop Height: 13'3
Object: To simulate a drop onto the bottom of the SNMC.	

## Appendix H:

December 2005 Baseline Results	Baseline run for White Masking Tape
Object: To determine the recovery capability of the White Masking Tape, and to determine how much of the recovery weight is from dust or pollutants.	

## Appendix I:

December 2005 Drop Test Results	Orientation: Side / Side Handle
Lid: 2-thread	Drop Height: 4'
Object: To simulate a drop onto the handles or side of a transfer vehicle.	

## Appendices, continued

### Appendix J:

December 2005 Drop Test Results	Orientation: Center of Gravity / Center
Lid: 2-thread	Drop Height: 4'
Object: To strike on edge of lid near filter.	

### Appendix K:

December 2005 Drop Test Results	Orientation: Bottom
Lid: 2-thread	Drop Height: 4'
Object: To strike on bottom.	

### Appendix L:

May 2007 Results

## Appendix A

<b>November 2005 Drop Test Results</b> <b>Lid: 2-thread</b> <b>Object: To strike on edge of lid near filter.</b> NOTE: After opening container, there was a consistent phenomenon of powder collected in the first two body threads. C. J. Mills <b>Data recorded by: J. A. Vargas</b>		<b>Orientation: Center of Gravity / Center</b> <b>Drop Height: 13'3</b>
Test Number	Size (in Quarts)	Results / Comments
1	?	No information written.
2	?	No information written.
3	?	No information written.
4	?	No information written.
5	3	<b>This test was a complete failure.</b> Lid completely popped off.
6	3	Visible release of powder, but the lid stayed on.
7	3	There was a minimal release of powder, The lid stayed on. Note: There is not much powder on the threads of the container.
8	8	Lid stayed on can with minimal release.
9	8	The lid stayed on the can. Release was minimal. The threads were partly damaged and the lid was difficult to remove.
10	8	There was minimal release of powder in the bag and around the threads of the can.
11	12	There was a minimal release of powder in the bag and on the threads of the can. NOTE: The container took some structural damage on the lid and just below the welded portion of the thread-ring.
12	12	The lid popped completely of this container and there was a definite release of powder. <b>Total failure.</b>
13	12	There was a minimal release of powder in the bag and around the threads of the body. NOTE: The can sustained the same structural damage as test #11.
14	5	This test showed a definite release of powder. The amount of powder was more than the rest of the tests that have not completely failed.
15	5	There was a minimal release of powder in the bag and around the threads.
16	5	There was a minimal release of powder, considering that the lid jumped a thread on the opposite side of impact. NOTE: The lid could not be removed.

## Appendix B

<b>November 2005 Drop Test Results</b> <b>Orientation: Side / Side-handle</b> <b>Lid: 2-thread</b> <b>Drop Height: 13'3</b> <b>Object: To simulate a drop onto the handles or side of a transfer vehicle.</b> NOTE: After opening container, there was a consistent phenomenon of powder collected in the first two body threads. C. J. Mills <b>Data recorded by: J. A. Vargas</b>		
<b>Test Number</b>	<b>Size (in Quarts)</b>	<b>Results / Comments</b>
17	1	This test was a <b>complete failure</b> . Major amounts of powder from a broken weld on the upper lip under the thread-ring above the side-handle. NOTE: This test was a side handle drop test dropped onto a 1-1/4" steel bar.
18	12	There was a minimal release of powder. NOTE: The impact was in the middle of the side-handle. The container was severely damaged on the side, but was not breached.

## Appendix C

<b>December 2005 Baseline Results</b> <b>Baseline run for Blue Masking Tape</b> <b>Object: To determine the recovery capability of the Blue Masking Tape, and to determine how much of the recovery weight is from dust or pollutants.</b> <b>Data recorded by: C. J. Mills</b>	
<b>Standard Test Number</b>	<b>Results / Comments</b>
1	Took empty bag and smeared with tape. Yielded 0.00060g
2	Placed a known amount of powder (0.00332g) in bag and shook bag. Recovered 0.00137g. Subtract 0.00060, and corrected recovery is 0.00077, which is a 43.7% recovery.

## Appendix D

December 2005 Drop Test Results				Orientation: Center of Gravity / Center	
Lid: 2-thread				Drop Height: 13'3	
Object: To strike on edge of lid near filter.					
NOTE: After opening containers, there was a consistent phenomenon of powder collected in the first two body threads.					
Data recorded by: C. J. Mills					
Test Number	Size (in Quarts)	Initial Load (g)	Recovered Puff (g)	Comments	Corrected Puff (g)
1	1	20.08		Impact was on filter. Visual release. Could not recover all of release. Difficulty weighing with cloth recovery method	Did not calculate
2	1	24.957		Some visual release. Difficulty weighing bag to get a tare weight.	Did not calculate
3	1	24.957		Visible powder. Difficulty weighing release on glove because of movement.	Did not calculate
4	3			Visual release. Couldn't get glove to weigh out.	Did not calculate
5	3	22.144	0.00084	Started using blue Masking Tape for recovery efforts.	Blue 0.00192
6	3	30.193	0.00410	Jumped a thread opposite the impact point. Hit on lid next to filter.	Blue 0.00938
7	5	26.421	0.00199	Hit on corner by filter	Blue 0.00455
8	5	29.460	0.00187	Hit on corner by filter	Blue 0.00428
9	5	24.300	0.00147	Modified body	Blue 0.00336
10	8	27.258	0.00220	Visible release. Hit on corner next to filter	Blue 0.00503
11	8	19.90232	0.00224	Visible release. Hit on corner next to filter	Blue 0.00513
12	8	20.004553	0.00385	Unscrewed lid by 1-1/4". Lid couldn't be moved. Visible release. Hit on corner next to filter	Blue 0.00881

## Appendix D, continued

<b>December 2005 Drop Test Results</b> <b>Lid: 2-thread</b> <b>Object: To strike on edge of lid near filter.</b> NOTE: After opening containers, there was a consistent phenomenon of powder collected in the first two body threads. <b>Data recorded by: C. J. Mills</b>				<b>Orientation: Center of Gravity / Center</b> <b>Drop Height: 13'3</b>	
Test Number	Size (in Quarts)	Initial Load (g)	Recovered Puff (g)	Comments	Corrected Puff (g)
13	12	21.18878	0.00420	Hit on corner next to filter.	Blue 0.00961
14	12	21.44052	0.00170	Hit on corner next to filter.	Blue 0.00389
15	12	21.69885	0.00480	Hit on corner next to filter.	Blue 0.01098
16	1	21.80415	0.00346	Visual release. Hit on filter.	Blue 0.00792
17	1	22.87220	0.00402	Visual release. Hit on filter.	Blue 0.00920
18	1	24.17445	0.00277	Visual release. Hit on filter.	Blue 0.00634

## Appendix E

<b>December 2005 Drop Test Results</b> <b>Lid: 4-thread</b> <b>Object: To strike on edge of lid near filter.</b> NOTE: After opening containers, there was a consistent phenomenon of powder collected in the first two body threads. <b>Data recorded by: C. J. Mills</b>				<b>Orientation: Center of Gravity / Center</b> <b>Drop Height: 13'3</b>	
Test Number	Size (in Quarts)	Initial Load (g)	Recovered Puff (g)	Comments	Corrected Puff
1	1	22.28325	0.00192	Recovered puff using blue Masking Tape. Hit on edge of filter. Very slight visible release.	Blue 0.00439
2	1	21.60050	0.00090	Hit on edge of filter. Visible release.	Blue 0.00206
3	1	21.30052	0.00248	Hit on edge of filter. Visible release.	Blue 0.00568
4	3	22.54147	0.00168	4-thread. No visible release. Hit on corner near filter	Blue 0.00384
5	3	22.35531	0.00268	4-thread. Visible release. Hit on corner near filter	Blue 0.00613
6	3	22.89080	0.00289	4-thread. Barely visible release. Hit on corner near filter	Blue 0.0066
7	5	23.03169	0.00312	4-thread. Hit on corner near filter	Blue 0.00714
8	5	21.07200	0.00273	4-thread. Barely visible release. Hit on corner near filter	Blue 0.00625
9	5	21.97286	0.00296	4-thread. Barely visible release but more than previous can. Hit on corner near filter	Blue 0.00677
10	8	21.08816	0.00484	4-thread. Visible release. Hit on corner near filter	Blue 0.01108
11	8	24.3654	0.00274	4-thread. No visible release. Hit on corner near filter	Blue 0.00627
12	8	28.69260	0.00489	4-thread. Visible release. Hit on corner near filter	Blue 0.01119

## Appendix E, continued

December 2005 Drop Test Results

Lid: 4-thread

Object: To strike on edge of lid near filter.

NOTE: After opening containers, there was a consistent phenomenon of powder collected in the first two body threads.

Data recorded by: C. J. Mills

Orientation: Center of Gravity / Center

Drop Height: 13'3

Test Number	Size (in Quarts)	Initial Load (g)	Recovered Puff (g)	Comments	Corrected Puff
13	12	21.73989	0.00321	4-thread. Hit on corner near filter	Blue 0.00735
14	12	23.82992	0.00227	4-thread. No visible release. Hit on corner near filter. Jumped a thread opposite the point of impact.	Blue 0.00519
15	12	23.00550	0.00497	4-thread. No visible release. Hit on corner near filter. ~ one dozen metal filings on tape.	Blue 0.01137

## Appendix F

<b>December 2005 Drop Test Results</b> <b>Lid: 2-thread</b> <b>Object: To simulate a drop onto the handles or side of a transfer vehicle.</b> NOTE: After opening containers, there was a consistent phenomenon of powder collected in the first two body threads. <b>Data recorded by: C. J. Mills</b>				<b>Orientation: Side / Side Handle</b> <b>Drop Height: 13'3"</b>	
Test Number	Size (in Quarts)	Initial Load (g)	Recovered Puff (g)	Comments	Corrected Puff
1	3	20.23236	0.00173	Impact 180 degrees from side handle. Hit on bottom of container. Visible release.	Blue 0.00400
2	3	23.27270		<b>Catastrophic event.</b> Hit just above side handle. Body deformed under lid.	
3	3			<b>Catastrophic event.</b> Impact under weld seam, 180 degrees from side-handle. Body deformed under lid. Significant release. Able to remove lid and found powder in first two body threads.	
4	5	22.57522	0.00245	Impact 180 degrees from side-handle. No visible release.	Blue 0.00561
5	5	23.91084	0.00249	Impact 180 degrees from side handle.	Blue 0.00570
6	8	31.22570	0.00360	We're getting a feel for where an impact needs to occur for a catastrophic event. We're trying to finalize these results. Impact 180 degrees from side-handle. Hit 4-1/2" from bottom. No visible release but recovered very fine powder.	Blue 0.00824
7	8	24.12699	0.00336	Impact 180 degrees from side-handle. No detectable release. Hit 6-3/4" from bottom	Blue 0.00769

## Appendix F, continued

December 2005 Drop Test Results				Orientation: Side / Side Handle	
Lid: 2-thread				Drop Height: 13'3	
Object: To simulate a drop onto the handles or side of a transfer vehicle.					
NOTE: After opening containers, there was a consistent phenomenon of powder collected in the first two body threads.					
Data recorded by: C. J. Mills					
Test Number	Size (in Quarts)	Initial Load (g)	Recovered Puff (g)	Comments	Corrected Puff
8	8			Catastrophic event. Hit 7-7/8” from bottom. Visible release. Deformed body under lid threads. Imagine a solid side-handle all the way around a container. If the impact occurs at the top of the side handle to the bottom of the lid, the container is vulnerable to deformation of the body under the lid or of the welds, in this region.	
9	1	20.93265	0.00214	Hit at top of side-handle at 2-3/4” from bottom. Very little release.	Blue 0.00490
10	1	20.22153	0.00239	Hit 180 degrees from side-handle at 2-3/4” from bottom. Visible release.	Blue 0.0055
11	1	21.22411		<b>Catastrophic event.</b> Changed to White Masking Tape. Hit opposite side handle, but we’re in the vulnerable area, at 3” from bottom.	
12	12	21.90636	0.00417	Hit halfway into side-handle. Some visible release. Impact site 6-3/4” from bottom	White 0.00463
13	12	20.10372	0.00508	No visible release. Hit toward top of side-handle. Hit at 8-1/4” from bottom.	White 0.00564
14	12	21.71526	0.00430	Hit right below collar weld (weld between collar extension and body) at approximately 10” from the bottom. 180 degrees from side-handle. High visible release.	White 0.00478

## Appendix G

<b>December 2005 Drop Test Results</b>				<b>Orientation: Bottom</b>	
<b>Lid: 2-thread</b>				<b>Drop Height: 13'3</b>	
<b>Object: To simulate a drop onto the bottom of the SNMC</b>					
NOTE: After opening containers, there was a consistent phenomenon of powder collected in the first two body threads.					
<b>Data recorded by: C. J. Mills</b>					
<b>Test Number</b>	<b>Size (in Quarts)</b>	<b>Initial Load (g)</b>	<b>Recovered Puff (g)</b>	<b>Comments</b>	<b>Corrected Puff</b>
7	1	20.17537	0.00399	Landed on bottom.	White 0.00443
8	12	21.91526	0.00470	Landed on bottom.	White 0.00522

## Appendix H

<b>December 2005 Baseline Results</b>		<b>Baseline run for White Masking Tape</b>	
<b>Object: To determine the recovery capability of the White Masking Tape, and to determine how much of the recovery weight is from dust or pollutants.</b>			
<b>Data recorded by: C. J. Mills</b>			
<b>Standard Test Number</b>	<b>Results / Comments</b>		
1	Took empty bag and smeared with tape. Yielded 0.00060g		
2	Placed a known amount of powder (0.00922g) in bag and shook bag. Recovered 0.00997g. Subtract 0.00060 and the corrected recovery is 0.00937. This is greater than a 100% recovery. To add a little conservatism, I will use 90% recovery.		

## Appendix I

<b>December 2005 Drop Test Results</b> <b>Lid: 2-thread</b> <b>Object: To simulate a drop onto the handles or side of a transfer vehicle.</b> NOTE: After opening containers, there was a consistent phenomenon of powder collected in the first two body threads. <b>Data recorded by: C. J. Mills</b>				<b>Orientation: Side / Side Handle</b> <b>Drop Height: 4'</b>	
Test Number	Size (in Quarts)	Initial Load (g)	Recovered Puff (g)	Comments	Corrected Puff
1	1	24.20235	0.00161	Will concentrate on trying to create a catastrophic event. Impact 180 degrees from side-handle and 3" from bottom in vulnerable area. No visible release	White 0.00179
2	1	20.25456	0.00385	Will concentrate on trying to create a catastrophic event. Impact 180 degrees from side-handle and right under lid in vulnerable area. No visible release	White 0.00428
3	3	20.28783	0.00430	This is getting boring. I can't seem to kill containers from a 4' drop, but we're still seeing the puff. Hit below lid. No visible release.	White 0.00478
4	3	20.44609	0.00557	Hit 5-1/2" from bottom	White 0.00619
5	8	23.81084	0.00415	Hit right below lid, at 9" from bottom. Visible release.	White 0.00461
6	8	22.64087	0.00778	Dropped bar on SNMC. Hit above side-handle. Visible release.	White 0.00864
7	12	20.84475	0.00803	Hit just below collar weld, 10" from bottom. Very slight visible.	White 0.00892
8	12	20.72205	0.00338	180 degrees from side-handle. Hit just under lid, on collar. No visible release	White 0.00376

## Appendix J

<b>December 2005 Drop Test Results</b>				<b>Orientation: Center of Gravity / Center</b>	
<b>Lid: 2-thread</b>				<b>Drop Height: 4'</b>	
<b>Object: To strike on edge of lid near filter.</b>					
NOTE: After opening containers, there was a consistent phenomenon of powder collected in the first two body threads.					
<b>Data recorded by: C. J. Mills</b>					
<b>Test Number</b>	<b>Size (in Quarts)</b>	<b>Initial Load (g)</b>	<b>Recovered Puff (g)</b>	<b>Comments</b>	<b>Corrected Puff</b>
1	1	22.39371	0.00234	Hit on filter.	White 0.00260
2	3	21.61239	0.00227	Hit on filter. No visible release	White 0.00252
3	8	20.88222	0.00571	Hit next to filter on edge. No visible release.	White 0.00634
4	12	21.32807	0.00426	Slight visible release. Hit on edge next to filter	White 0.00473

## Appendix K

<b>December 2005 Drop Test Results</b>				<b>Orientation: Bottom</b>	
<b>Lid: 2-thread</b>				<b>Drop Height: 4'</b>	
<b>Object: To strike on bottom.</b>					
NOTE: After opening containers, there was a consistent phenomenon of powder collected in the first two body threads.					
<b>Data recorded by: C. J. Mills</b>					
<b>Test Number</b>	<b>Size (in Quarts)</b>	<b>Initial Load (g)</b>	<b>Recovered Puff (g)</b>	<b>Comments</b>	<b>Corrected Puff</b>
1	1	20.17537	0.00399	No visible release.	White 0.00443
2	12	21.91526	0.00470	No visible release.	White 0.00522



LA-UR- 99 - 3709

Title:

Nuclear material Container Testing

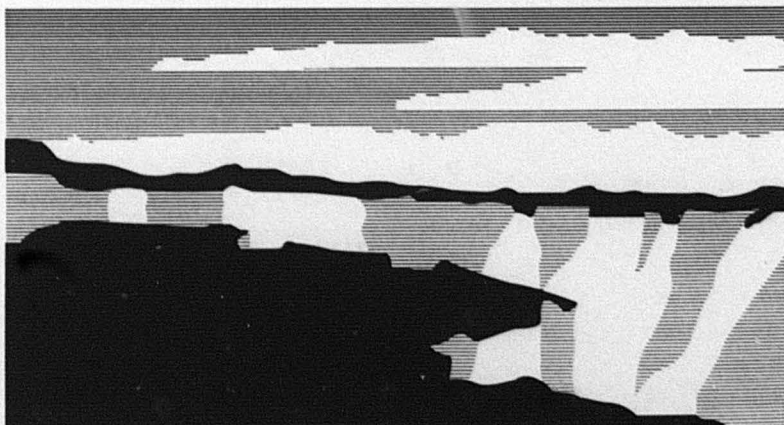
Author(s):

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**Los Alamos**  
NATIONAL LABORATORY



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## NUCLEAR MATERIAL CONTAINER TESTING

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### Abstract

The Los Alamos Nuclear Materials Technology Division (NMT) in collaboration with Nuclear Filter Technology, Inc. [1] (a commercial supplier) has fabricated and tested new nuclear material containers. The containers are designed for daily use, safe transport within the facility, and storage up to twenty years. They are outer containers used in a layered packing configuration with the nuclear material in an inner package. These containers are not used outside the facility. The Facility has invested over a million dollars in the design, testing and procurement of these containers and has them in local stock for routine use. Los Alamos evaluated the effectiveness of the previous storage containers and found that many had safely contained nuclear material but that a limited number had failed. The results of a three-year study established the design requirements for the new containers. They must be easily opened/closed, not allow pressurization, be fabricated with 304 stainless steel, allow all nondestructive assay methods to be used, be easy to decontaminate, assure high reliability for containment, maintain a leak tight seal if dropped, and meet safeguards and criticality requirements. The containers are available in five sizes, from one to twelve quarts, and nest one into another. This paper describes the design and quality assurance requirements and the results of an extensive testing program used to qualify the nuclear material containers.

### Introduction

The Nuclear Materials Technology (NMT) Division at the Los Alamos National Laboratory (LANL) is responsible for the research and development of nuclear materials and is accountable for the nuclear materials in process and in storage. LANL's response to the Defense Nuclear Facility Safety Board directive 94-1 [2] requires continual handling, nondestructive assay and repackaging of these materials. Legacy materials from past programs dating back to the Manhattan Project in the 1940's exist in storage today. These materials are stored for their strategic value and because of their hazardous nature.

## Background

After a 3-year study that evaluated more than 2,000 storage items, we discovered a limited number of the containers used in storage had failed over time. Each item was opened, inspected, and in some cases repackaged into a safer configuration and/or container. An inspection sheet was filled out for each item, failures were often photographed and a database was established to assure adequate documentation for this project. Design criteria for a reliable storage container was created from the information gathered from this study. Nuclear Materials Technology Groups were asked to designate a person to give ideas, advice, and information concerning the use and storage of containers.

## Design Criteria

The most important container design concern is user safety. The containers must be robust, have few parts, and no sharp edges to cut or puncture gloves. They need to be easy to use and decontaminate for reuse. A vent was mandatory to prevent gas buildup and pressurization. Containers had to fit through existing bag-out ports and nest one into another for over-packing. Existing nondestructive assay techniques were to be used without modification and the containers had to meet safeguards and criticality requirements. Our study demonstrated that tin plated containers could corrode over time. 304 stainless steel was designated as the material for the body and lid because no problems were encountered with items that had been stored for several years in stainless steel dressing jars. A positive closure lid was a requirement to assure a good seal because many of the failures had occurred with lids that were taped closed. Over time the tape can degrade and create a possible contamination problem. (Table 1 for Design Criteria.)

TABLE 1

### DESIGN CRITERIA

1	Set of four containers ranging in nominal size from one to twelve quart capacities.
2	Each container shall nest into the next larger size, and have a clearance of about two inches on the height and about ½ inch on the diameter.
3	Containers to be 304 stainless steel and have a body wall thickness of 0.020-0.032 inch. to allow transmission of radiation for nondestructive assay.
4	Container body shall be seamless, have a flat bottom with rounded inner edges, and a 32 standard finish (easy to wipe clean to allow decontamination).
5	The lids shall be of 304 stainless steel and have a positive closure mechanism such as a clamp or screw.
6	The lid shall be water tight sealed with an o-ring or rubber gasket.
7	The lid must contain a nuclear materials filter similar to the Nuclear Filter Technology Inc. model NUCFIL-030.
8	The assembled containers loaded to ½ volume with dry sand must withstand a nine-foot drop impacting on concrete (flat on bottom, inverted, side of body, 45° on lid). The container must not rupture or leak. Deformation of the container is expected. A test will be performed by pressurization through the filter hole to 2 psi with the container submerged in water. Container will pass if no bubbles are observed.

### Vendor Selection for Container Fabrication

The design criteria were sent to twelve companies requesting their designs to meet our specifications. Three companies sent drawings and two companies agreed to make prototypes for our evaluation. Prototypes were ordered for evaluation from the two companies. Each set of prototypes met our design criteria. Experienced technicians assessed each set of containers and selected the container design we are now using because it was easier to open and close and had fewer parts (see containers with handles in Figure 1). Nuclear Filter Technology, Inc. is the manufacturer of our nuclear material containers. Tests were performed on the container to assure reliability before procurement.

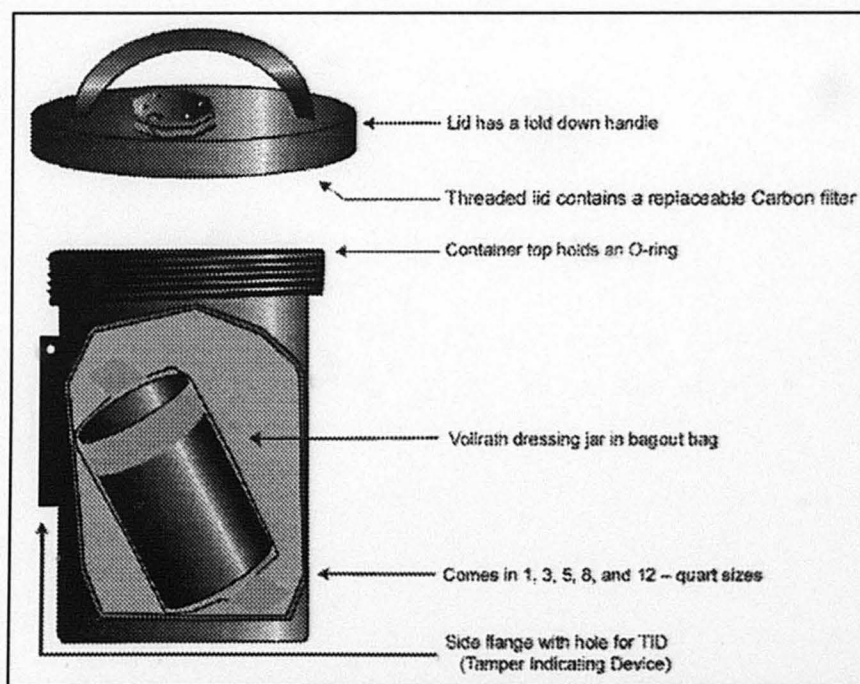
FIGURE 1



### Additional Features

Additional features were added to the containers at our request. The filter can be removed and replaced if necessary and the lids and bodies are interchangeable. LANL's Operational Security required a tamper-indicating device (TID) on the filter. A TID wire is welded from the filter to the container lid to enhance safeguards. The raised filter minimizes possible water entry through the Gore-Tex liner. A fold down handle replaced the rigid handle on the container. Containers have a side flange, which may be used to secure a new shielding over-pack and aid in opening and closing the container. The side flange on the body of the container contains a hole to attach a cable style TID to the lid. (Figure 2)

FIGURE 2



## **Initial Drop Tests**

To verify the containment capability of the container, LANL and the manufacturer conducted drop tests followed by qualitative leak tests. LANL performed drop tests at a height of eight feet with a loaded container in four orientations with bubble tests before and after each drop. Drop orientations were difficult to maintain on the containers. Modifications to the original design were made as indicated by the testing results. Drop tests were conducted with a loaded container by the manufacturer at a height of four feet and eight feet onto an unyielding surface in five orientations; on the handle, bottom, side, and 45° on top and bottom to verify leak tightness under impact. After the drop test the container was pressurized to 5" water column differential and submerged in water to check for leakage. Nuclear Filter Technology, Inc. and Rocky Flats recorded the results of the drop tests [3]. Testing results verified a reliable container and they are now in use at LANL and Rocky Flats. Later, Verification and Certification by an independent division at LANL to determine vulnerabilities in the new container was recommended.

## **Manufacturers Testing, Qualification and Records**

Each filter is leak and efficiency tested by the manufacturer. A hydrogen diffusivity test is conducted to verify that filter diffusivity characteristics exceed the WIPP requirements for venting filters. Gore-Tex liners over the filters are individually pressure tested at 40" to 50" column pressure to verify that there is no water entry. The filters are WIPP certified for Nuclear Materials. The containers are leak tested, serialized and checked for interchangeability (two sets of master containers were fabricated by the manufacturer for test purposes, one set was retained by the manufacturer, the other by LANL). The filter and containers are manufactured and tested according to the manufacturers Quality Assurance (Q.A.) Program. Nuclear Filter Technology developed their Q.A. Manual to meet requirements contained in ASME NQA-1-1989, N45.2-1986 [4]. The manufacturer retains records concerning testing and qualification of the containers and filters.

## **LANL Testing and Records**

Nuclear Materials Technology (NMT) personnel perform Quality Assurance tests on up to 10% of the containers in each shipment at the manufacturer's plant before they are shipped to LANL. Containers are selected at random, the welded TID wire is removed from the filter, and a leak test and inspection is performed (Table 2). Upon arrival at LANL 100% of the shipment receives a visual inspection as described in Table 3. Up to 10% of the shipment is helium leak tested (quantitative measurement result). Water intrusion tests are also performed. If a container fails any of the tests, 100% of the containers are then tested and all failures rejected. Records are retained on all Q.A. tests, inspections, and documentation. A permanent database is also retained with the same information. Containers can be cross-referenced using serial numbers retained in the database if necessary.

TABLE 2

**CONTAINER QA TESTS PERFORMED BY LANL  
AT THE MANUFACTURER'S FACILITY**

1	Interchangeability, all parts with the master container.
2	Height within $\pm 0.2$ inch
3	Diameter within $\pm 0.2$ inch.
4	Free of burrs and defects.
5	Labels are correct.
6	Containers are clean
7	Lid and body passes pressure test
8	Container pass or fail
9	Inspector initials the inspection form.

TABLE 3

**CONTAINER FINAL ACCEPTANCE TESTS AT LANL**

1	Container serial number is recorded.
2	Filter number is recorded.
3	Visual inspection verifies no container damage.
4	Labels in place and correct.
5	Tamper indication wire welded in place.
6	Containers pass or fail.
7	Inspector initials the final inspection form.

### Verification and Certification Drop Tests

Drop tests were designed and conducted at LANL's Engineering Science Applications-Measurement Technology (ESA-MT) at LANL by a Materials Engineer. A helium leak test was performed on each container before and after the drop test to give us a quantitative pass/fail. Containers have the same wall and lid thickness but vary in size: twelve, eight, three and one quart. The Materials Engineer recommended the testing focus on the largest (most vulnerable) container. Each drop orientation was tested with the largest container (twelve-quart) first. If it failed, the next size smaller was tested. If a container passed the drop test, no further testing was necessary in that drop orientation. The initial drop test height was eight feet and if each size container failed that orientation, they were then tested at a six-foot drop in the same orientation (starting with the twelve-quart can). The containers were loaded and dropped in seven impact orientations. The Materials Engineer rigged the containers on guidelines to assure the drop was in the correct orientation. The test passed if the container had an after drop leak rate of  $1 \times 10^{-5}$  or better. The acceptable leak rate will not allow the minimum particle size contamination to pass through a leak hole (we use the same criteria used to test glove boxes before they are approved for use with nuclear materials). Each drop test was videotaped and/or captured by still photos. Notes and results were recorded. The results of the LANL drop tests are shown in Table 4.

TABLE 4

## KEY TO TEST CODE

1F8 ie. NMC-12 can, upright impact, dropped 8 foot.

## CONTAINERS

1 =NMC-12 can  
 2 =NMC-8 can  
 3 =NMC-3 can  
 4 =NMC-1can  
 5=NMC2-5 can

## Drop configuration (impact)

A=Inverted impact  
 B=45 degree lid  
 C=90 degree Side on Body  
 D=Parallel Flange Shear  
 E=Base of Lid threads  
 F=Upright impact  
 G=90 degree Side Flange

## DROP DISTANCE

4 = 4' drop  
 6 = 6' drop  
 8 = 8' drop

## REPEAT THE TEST

none= first test  
 a =second test  
 b =third test  
 c = fourth test

BODY ID	TEST Code	TOTAL WT.	CONTACT		PRE-DROP LEAK RATE	AFT-DROP LEAK RATE	PASS/FAIL REPEAT	
			AREA OF CAN				R/P/F	TEST DATE
120043	1F8	11.001 kg.	bottom		1x10-6	1x10-6	P	1/6/99
120034	1A8	11.000 kg.	handle		1x10-6		F	1/6/99
800019	2A8	6.999 kg.	handle		sniffer		Repeat	1/7/99
80115	2A8a	7.013 kg.	handle		8x10-7	1.6x10-6	P	1/7/99
120038	1D8	11.000 kg.	flange		1x10-6	1x10-6	P	1/7/99
120028	1A8a	11.001 kg.	handle		1x10-6	1x10-6	P	1/7/99
120046	1D8a	11.001 kg.	flange		1x10-6	1x10-6	P	1/7/99
80109	2D8	7.000 kg.	flange		1x10-6	1x10-6	P	1/7/99
80145	2F8	7.000 kg.	flat bottom		1x10-6	1x10-6	P	1/7/99
120026	1E8	11.000 kg.	top		1x10-6	1x10-6	Repeat	1/11/99
120030	1E8a	10.999 kg.	lid bottom		1x10-6		F	1/11/99
120027	1B8	11.002 kg.	45 d. on lid		1x10-6	1x10-6	P	1/11/99
120042	1C8	11.001 kg.	90 d. body		1x10-6	1x10-2	F	1/11/99
120049	1G8	10.999 kg.	90 d. flange		1x10-6		F	1/11/99
80124	2B8	7.003 kg.	45 d. on lid		4x10-8	5x10-7	P	1/11/99
80123	2E8	7.001 kg.	15 d. on lid		3x10-7	2x10-8	P	1/11/99
10141	4C8	2.000 kg.	90 d. body		2.4x10-7		F	3/2/99
80200	2G8	7.001 kg.	90 d. flange		5x10-8		F	3/2/99
30282	3C8	2.999 kg.	90 d. body		4.8x10-8		F	3/2/99
80202	2C8	7.000 kg.	90 d. body		5.8x10-8		F	3/2/99
30280	3G8	3.003 kg.	90 d. flange		5x10-8	5x10-8	P	3/2/99
80203	2B8	7.001 kg.	45 d. lid		5x10-8	5x10-8	P	3/2/99
12058	1C6	10.999 kg.	90 d. body		6x10-8		F	3/2/99
80216	2C6	7.001 kg.	90 d. body		5x10-8		F	3/2/99
30281	3C6	3.000 kg.	90 d. body		4.4x10-8		F	3/2/99
10139	4C6	2.000 kg.	90 d. body		9x10-8	1x10-7	P	3/2/99
NMC2-012-2	1C8a	10.999 kg.	90 d. body		5x10-8		F	6/3/99
NMC2-08-2	2C8a	7.000 kg.	90 d. body		4x10-8		F	6/3/99
NMC2-05-1	5C8	4.502 kg.	90 d. body		4x10-8		F	6/3/99
NMC2-03-1	3C8a	3.000 kg.	90 d. body		3.8x10-8	4x10-8	P	6/3/99

## Conclusion

Most of the 90° side of body orientation drops breached the weld next to the threads, pulling them away from the lid and breaking the seal between the lid and body of the can. The drop test failures in this orientation caused us to strengthen the area below the lid to maintain a leak free seal. After consultation, a collar was added to the one, three, and eight-quart containers and lengthened on the twelve-quart container. The collar was welded inside at the top and outside on the bottom (new generation containers). A new five-quart container was being added to our inventory at LANL so it was fabricated with the double weld collar (NMC2-05-1). The new generation containers with double weld collars were then dropped in the 90° side of body orientation as before (NMC2-012-2, NMC2-08-2, NMC2-05-1 and NMC2-03-1). The new generation three-quart container passed the drop test at eight feet; the larger containers failed (see Table 4). The new generation container proved to be superior to the original container. The cost of the new generation container will be substantial due to the extra fabrication and welding. LANL is evaluating cost versus gain and will make a decision to convert to the new container or continue to use the existing container in the future. Whether we use the original nuclear material container or the new generation container, we have a safer, easy to use container with many innovative features.

## References

- [1] Nuclear Filter Technology, Inc., 741 Corporate Circle, Suite R  
Golden Colorado, 80401
- [2] Defense Nuclear Facility Safety Board Recommendation 94-1 to the Secretary of  
Energy, May 26, 1994.
- [3] Testing and Validation of Threaded Lid Vented Nuclear Materials Storage  
Containers, Terry J. Wickland, Nuclear Filter Technology, Inc.  
Martin Mataya, Safe Sites of Colorado Rocky Flats Environmental Technology  
Site, Patram 98, Paris, France, Spring 1998.
- [4] Nuclear Filter Technology, Inc.  
Filter Efficiency Test Procedure (P.S. 6.0)  
Hydrogen Diffusivity Measurement Procedure (P.S. 26.0)  
Semi Permeable Filter Water Entry Test (P.S. 29.0)  
Pressure Test and Marking of NMC Containers (P.S. 28.0).

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## Test Report on Testing of Los Alamos National Laboratory On-site Storage Containers to DOE Draft Manual 441.1-1 Criteria

Revision 0  
November **XX**, 2009



ORNL-27 (4-00)



National Transportation  
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ORNL/NTRC-033  
Rev. 0

Global Nuclear Security Technology Division

## **Test Report on Testing of Los Alamos National Laboratory On-site Storage Containers to DOE Draft Manual 441.1-1 Criteria**

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## Test Report on Testing of Los Alamos National Laboratory On-site Storage Containers to DOE Draft Manual 441.1-1 Criteria

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## Leak Testing of Los Alamos National Laboratory On-site Storage Containers to DOE Draft Manual 441.1-1 Criteria

### EXECUTIVE SUMMARY

The Department of Energy's (DOE's) Office of Nuclear Safety Policy and Assistance (HS-21) collaborated with the Los Alamos National Laboratory (LANL) and Oak Ridge National Laboratory (ORNL) to conduct container leak tests to evaluate and demonstrate methods for testing nuclear material container packages in support of DOE efforts to develop a final DOE Manual 441.1-1, *Nuclear Materials Packaging Manual*, and to evaluate the capability of some container types to meet the draft Manual requirements. On September 4 to 11, 2007, pre- and post-drop leakage rate tests were performed on ten containers at the National Transportation Research Center by ORNL's Transportation Technologies Group (TTG). The test results demonstrated the feasibility of methods for testing containers to the draft Manual 441.1-1 leakage criteria and demonstrated that some of the existing LANL container types used to store nuclear material could not meet all of the proposed leak rate criteria, in particular the post drop test leak rate criteria.

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## 1 PURPOSE

The Department of Energy's (DOE's) Office of Nuclear Safety Policy and Assistance (HS-21) collaborated with the Los Alamos National Laboratory (LANL) and Oak Ridge National Laboratory (ORNL) to conduct container leakage rate tests to evaluate and demonstrate methods for testing nuclear material container packages in support of DOE efforts to develop a final DOE Manual 441.1-1, *Nuclear Materials Packaging Manual*. These tests were also conducted to provide insight into whether the LANL container types could meet the requirements contained in the September 2007 draft version of the Manual.

## 2 BACKGROUND

Draft DOE Manual 441.1-1 provides criteria for the design of containers used to store (on site) nuclear material in quantities greater than the Department of Transportation  $A_2$  values<sup>1</sup>. The purpose of the Manual's requirements is to ensure that a suitable engineered confinement barrier is provided to protect workers from release of material during storage and handling.

The draft Manual includes the following acceptance criteria for *design release rate* and *design qualification release rate*.

Design Release Rate: One of the following testable release rates must be utilized.

- Utilize ANSI N14.5-1997 criteria for leaktight.

ANSI N14.5-1997 defines leaktight as:

“A degree of package containment that in a practical sense precludes any significant release of radioactive materials. This degree of containment is achieved by demonstration of a leakage rate less than or equal to  $1 \times 10^{-7}$  ref·cm<sup>3</sup>/s, of air at an upstream pressure of 1 atmosphere (atm) absolute (abs) and a downstream pressure of 0.01 atm abs or less.”

- Utilize 10 CFR § 71.51 criteria for no loss of radioactive material.

10 CFR § 71.51 specifies that for normal transport of radioactive material there should be no loss or dispersal of radioactive contents--as demonstrated to a sensitivity of  $10^{-6}$   $A_2$  per hour.

Design Qualification Release Rate Performance Objective. The package must have a post-drop design qualification release rate that will prevent the exposure of workers to greater than 5 rem CEDE. The drop test must be from the maximum working or storage height but not less than four feet. An acceptable value for the design qualification release rate is less than  $10^{-3}$   $A_2$ /event.

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<sup>1</sup>  $A_2$  values are defined in 49 CFR 171.435

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## 3 TEST OVERVIEW

### 3.1 Test Containers

Ten LANL containers were tested by the ORNL Transportation Technologies Group (TTG) to demonstrate compliance with selected requirements of Draft DOE Manual 441.1-1. The following LANL containers were tested:

- 8-quart, 4-thread LANL Special Nuclear Material Containers (SNMCs) (4 total),
- 8-quart, 2-thread LANL SNMCs (2 total),
- 5-gallon ring-closure drum (2 total), and
- 10-gallon ring-closure drum (2 total).

These containers (both with and in some cases without surrogate payloads) served as test units and were identified by sequential designation numbers TU-1-8/07 through TU-10-8/07. Hereafter in this test report, the date 8/07 will be dropped from the identification number of each test unit. Figure 3-1 shows an example of an 8-quart SNMC container.

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Test unit designations for each type of container are provide below in Table 3.1 shows an example of a container test unit

. Details of each Test Container's design are provided in Appendix 1.

**Table 3.1 Test Unit Description and Designations**

<b>Container Description</b>	<b>Test Unit Designation</b>
8-qt, 4 thread SNMC Container	TU-1, TU-2, TU-3, TU-4
8-qt, 2 thread SNMC Container	TU-5, TU-6
5-gal Ring Closure Drum	TU-7, TU-8
10-gal Ring Closure Drum	TU-9, TU-10

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Figure 3-1 TU-1 (8-quart, 4-thread, SNMC) prior to testing

## 3.2 Test Procedures

One test unit from each container type was first assembled empty, subjected to a helium leak test, and then disassembled. All test units were then loaded with a payload (tungsten shot in taped slip lid cans), assembled, subjected to a water-bubble leak test, drop tested, and again subjected to a water-bubble leak test. Details of the testing sequence for each test unit are provided in Chapters 4 and 5.

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## 3.3 Test Data

This report documents the tests performed and measurements observed. The general data types for these tests are.

- Procedure checklist
- Manually derived measurements and observations documented in data sheets
- Digital still photography
- Videographs of drop tests

The completed Data Sheets and Procedure Checklists have been scanned into digital form and are included as Appendix 3 of this report.

## 3.4 Test Acceptance Criteria

One of the purposes for performing the testing was to develop the test acceptance criteria that corresponds to the Manual criteria accounting for the testing equipment and method utilized. The procedure for translating the draft Manual acceptance criteria into a measurable leak rate criteria (both a He leak test and air bubble test rates) outlined in ANSI N14.5-1997 was utilized. The details of the procedure are provided in LANL Report LA-UR-08-06823, "Design Leak Rates for Plutonium Containers", 2008. The resulting test acceptance criteria for the He and air leak rates are a function of (1) differential pressure utilized in the test and (2) assumed radioactive material being tested (non-radioactive surrogate material was utilized for these tests). The results for the Manual's design leak rate criterion ( $10^{-6}$  A<sub>2</sub> per hour) tested utilizing He and design qualification leak rate criteria ( $10^{-6}$  A<sub>2</sub> per event) tested utilizing air are shown in Table 3.2 Test Acceptance Criteria below:

**Table 3.2 Test Acceptance Criteria**

Test Criteria/Medium	Differential Pressure	Material Type	Test Acceptance Criteria							
Design Leak Rate/He	1 kPa	Weapons Grade Pu			$6.8 \times 10^{-5}$ atm cm <sup>3</sup> /s					
Design Leak Rate/He	1 kPa	Heat Source Pu			$1.3 \times 10^{-6}$ atm cm <sup>3</sup> /s					
Design Leak Rate/He	2 kPa	Weapons Grade Pu			$8.5 \times 10^{-5}$ atm cm <sup>3</sup> /s					
Design Qualificati	2 kPa	Weapons Grade Pu			$8.5 \times 10^{-5}$ atm					

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on Leak Rate/Air			cm <sup>3</sup> /s							
Design Qualificati on Leak Rate/Air	5 kPa	Weapons Grade Pu	29 cm <sup>3</sup> /s							
Design Qualificati on Leak Rate/Air	5 kPa	Heat Source Pu	.22 cm <sup>3</sup> /s							
Design Qualificati on Leak Rate/Air	5 kPa	Drum??	19 cm <sup>3</sup> /s							

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## 4 DESIGN RELEASE RATE TESTING

### 4.1 Test Procedure

A Varian 959 Turbo Helium Leak Detector was utilized to determine the Design Release Rate of a representative of each container type. The container was placed in a vacuum chamber and the container and chamber were evacuated simultaneously to a vacuum of 100 milliTorre or less (Figure 4-1 is a schematic of the test apparatus, Figure 4-2 is a picture of the test apparatus connect to Test Unit 1). The Test Unit and the vacuum chamber were then isolated from one another by closing the Valve #1 in Figure 4-1, below. Helium was then introduced into the Test Unit (container) to a prescribed pressure (approximately 1 kPa for Test Units 1 and 5, and 2 kPa for Test Units 7 and 9) through the use of Valve #2. The vacuum is pulled through the helium leak detector such that any leakage of helium from the Test Unit into the vacuum chamber is detected by the leak detector. The leak rate is recorded at 2 minute intervals for 20 minutes or until the leak rate is off scale on the leak detector. These tests were performed according to TTG Test Procedure TTG-PRF-02, Rev. 1, *Standard Full Boundary Leak Test Method – Helium Leak Testing*.

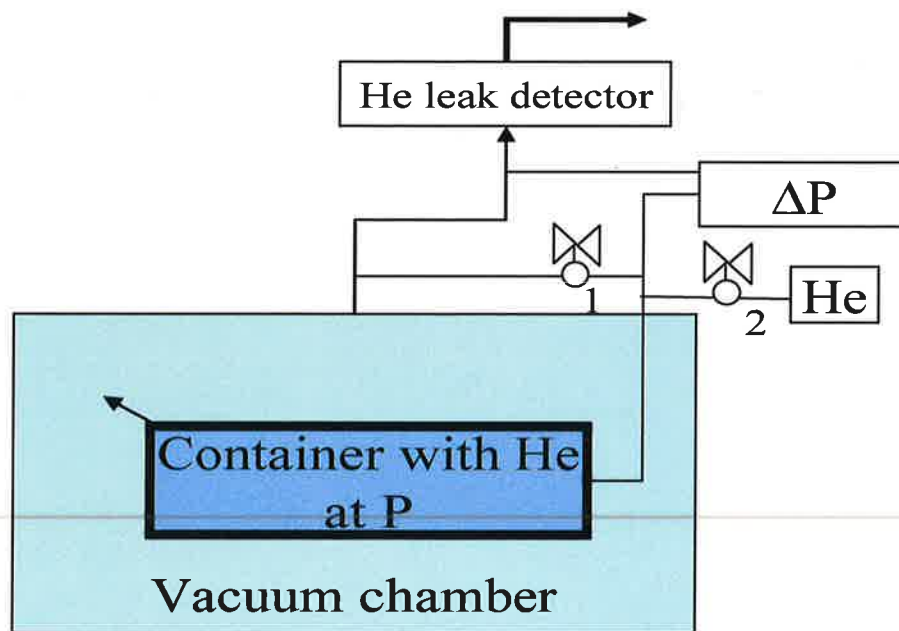


Figure 4-1 Helium leak detection system schematic

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A key parameter design release rate testing is the vacuum pulled on the Test Units. This is maintained as close to the target values (1 or 2 kPa depending on the test) as possible. A key variable identified during the test was the lid torque for the SNMC containers and tightness of the closure ring (as measure by the lug gap and ring gap) for the drums. This was not a parameter that was identified in the LANL specifications for the SNMC containers. Rather, the container design developed the degree of closure through the use of closure alignment markings on the lid and the container. The impact of this variable is discussed in the test results.

The test plan had called for a dwell time between test unit assembly and helium leakage rate testing of 72 hours; however, such a long dwell time was determined to not be required. The actual dwell time from assembly to initiation of the helium leak testing process (vacuum initiation) was shortened to 49.25 hours because vacuum measurements of the test unit assembly were unchanged after the first hour of placement of the Test Unit in the test chamber.

## 4.2 Test Results

Copies of the completed check lists and data sheets from these tests are contained in Appendix 3 of this report. Table 4.1 provides a summary of the Design Release Rate (He Leak Test) test results. Note that since a primary purpose of the testing was to determine and demonstrate an appropriate test method, some changes were made to the test procedure during the testing process. The details of the actual tests performed on each Test Unit exposed to helium leak testing are provided in the Table 4.1 below:

**Table 4.1 Design Release Rate (Helium Leak) Test Results**

Test	Test Unit	Lid Torque (in-lbs)	Test ΔP (kPa)	Measured Final Leak Rate (ccHe/s)	Reference Final Leak Rate (ref-cc Air/s)	Meets Criteria?/ Comments
1	TU-1 (SNMC 8-qt 4-thread)	100	1.20	$8 \times 10^{-5}$	$1.7 \times 10^{-4}$	Failed
1, Retest 1	TU-1	100	Test aborted	Test aborted	Test aborted	Test aborted
1, Retest 2	TU-1	100	1.11	$1.0 \times 10^{-7}$	$5.3 \times 10^{-8}$	Passed
2	TU-1	140	1.08	$> 1.0 \times 10^{-4}$	$> 1.0 \times 10^{-4}$	Failed/Test stopped
2, Retest 1	TU-1	140	1.32	$1.0 \times 10^{-7}$	$5.3 \times 10^{-8}$	Passed
3	TU-1	200	Test stopped	$> 1.0 \times 10^{-4}$	$> 1.0 \times 10^{-4}$	Failed/Test stopped
3, Retest 1	TU-1	200	2.72	$1.2 \times 10^{-7}$	$6.5 \times 10^{-8}$	Passed
4	TU-5 (SNMC 8-qt 2-thread)	140	1.13	$8.0 \times 10^{-8}$	$4.1 \times 10^{-8}$	Passed
5	TU-7 (5-gal drum)	Lug gap 5/16" Ring gap 7/16"	~0.15	$> 1.0 \times 10^{-4}$	$> 1.0 \times 10^{-4}$	Failed/Test stopped
6	TU-9 (10-gal drum)	Lug gap 1-1/8" Ring gap 9/16"	0.37	$2.4 \times 10^{-5}$	$5.0 \times 10^{-5}$	Failed

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## 4.2.1 Test Unit 1 (TU-1) (8-quart, 4-thread SNMC )Test Results

The lid of TU-1 was initially torqued to 100 in-lbs. 100 in-lbs was selected in the process of tightening down the lid, as a good closure and mating of the surface between the lid and the container was observed. After initial assembly of TU-1 a helium leak test was performed. The leak rate of TU-1 was  $> 1.0 \times 10^{-5}$  cc He/s in less than 2 minutes from test initiation. At 2 minutes into the test the leakage rate was  $2.0 \times 10^{-5}$  cc He/s, and steadily rose over the course of the test to a final leak rate of  $8.0 \times 10^{-5}$  cc He/s after 22 minutes of testing. The pressure difference between the vacuum chamber and the TU-1 was steady between 1.19 and 1.20 kPa throughout the duration of the test.

An attempt to repeat the helium leak test of TU-1 was aborted prior to reaching full vacuum conditions because the leak rate of TU-1 was ramping up past  $2.0 \times 10^{-5}$  cc He/s, and steadily rose over the course of the start of test vacuum pulldown.

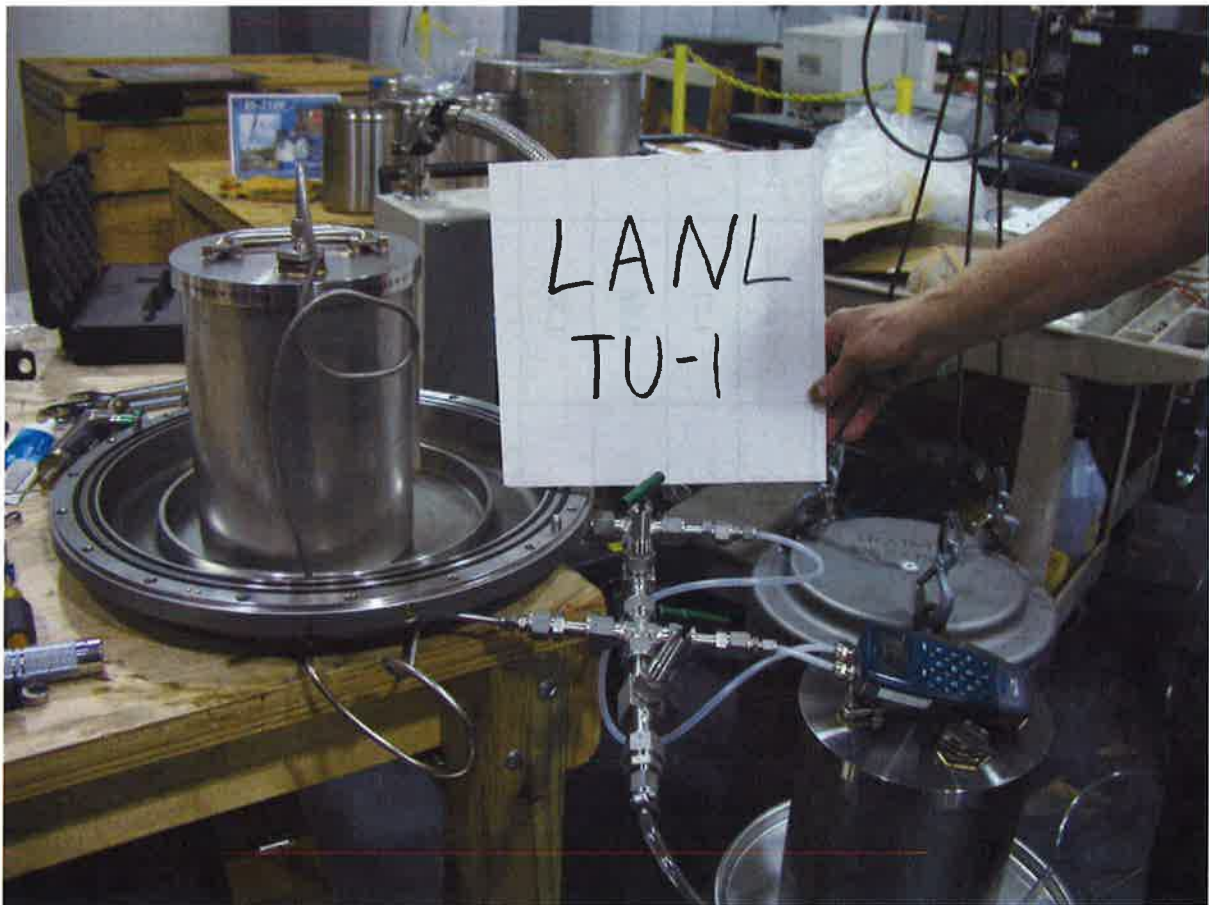


Figure 4-2 TU-1 being attached to the helium leak detection apparatus

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TU-1 was subsequently disassembled, the O-ring replaced, reassembled (empty), again with a closure torque of 100 in-lbs, and helium leak tested. At 2 minutes into the test the leakage rate was  $1.8 \times 10^{-8}$  cc He/s, and steadily rose over the course of the test to a final leak rate of  $1.0 \times 10^{-7}$  cc He/s after 20 minutes of testing. The pressure difference between the vacuum chamber and TU-1 was steady at 1.11 kPa throughout the duration of the test. TU-1 was subsequently disassembled and reassembled (empty) with a closure torque of 140 in-lbs. When a helium differential pressure of 1.08 kPa was applied to the test unit, the leak rate was immediately off scale on the helium leak tester (i.e. leak rate of  $> 1.0 \times 10^{-4}$  cc He/s). TU-1 was subsequently removed from the test apparatus and the lid removed. Lid removal required a torque of 130 in-lbs. TU-1 was then disassembled and reassembled with a closure torque of 200 in-lbs. When a helium differential pressure was applied to the test unit, the leak rate was immediately off scale on the helium leak tester (i.e. leak rate of  $> 1.0 \times 10^{-4}$  cc He/s).

The results of these first two tests on TU-1 suggested there may be a leak within the test apparatus. The test assembly system was inspected for leaks and valve connections were examined, reassembled with attention to the application of thread sealant and torque values on the valves. The reassembled test assembly system was then used to successfully test TU-1 again, as described below.

TU-1 was subsequently disassembled and reassembled with a new O-ring (lubricated with Parker Super O-lube), and a closure torque of 140 in-lbs. The test unit was then allowed to sit for 71.25 hours and then subjected to a helium leakage rate test. At 2 minutes into the test the leakage rate was  $1.6 \times 10^{-9}$  cc He/s, and steadily rose over the course of the test to a final leak rate of  $1.0 \times 10^{-7}$  cc He/s after 26 minutes of testing. The pressure difference between the vacuum chamber and TU-1 was steady at 1.32 kPa throughout the duration of the test. TU-1 was subsequently disassembled and reassembled with a new O-ring (lubricated with Parker Super O-lube), and a closure torque of 200 in-lbs. The test unit was subsequently subjected to a helium leakage rate test. At 2 minutes into the test the leakage rate was  $9.6 \times 10^{-9}$  cc He/s, and steadily rose over the course of the test to a final leak rate of  $1.2 \times 10^{-7}$  cc He/s after 22 minutes of testing. The pressure difference between the vacuum chamber and the TU-1 was steady at 2.72 kPa throughout the duration of the test.

## 4.2.2 Test Unit 5 (TU-5) (8-quart, 2-thread SNMC) Test Results

The lid of TU-5 was initially torqued to 140 in-lbs. After initial assembly of TU-5 a helium leak test was performed. The leak rate of TU-5 was  $1.0 \times 10^{-8}$  cc He/s two minutes after test initiation. The leakage rate steadily rose over the course of the test to a final leak rate of  $8.0 \times 10^{-8}$  cc He/s after 22 minutes of testing. The pressure difference between the vacuum chamber and TU-1 was steady at 1.13 kPa throughout the duration of the test.

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## 4.2.3 Test Unit 7 (TU-7) (5-gallon drum) Test Results

TU-7 was assembled according to LANL TA-55 Work Instruction TA55-WI-034, Revision 0. After closure, the lug gap on the closure ring was 5/16" and the ring gap was 7/16". An attempt to perform a helium leak test resulted in the helium leak detector going off-scale (i.e., leak rate  $> 1.0 \times 10^{-4}$  cc He/s) after about 15 seconds with a differential pressure of about 0.15 kPa applied.

## 4.2.4 Test Unit 9 (TU-9) (10-gallon drum) Test Results

TU-9 was assembled according to LANL TA-55 Work Instruction TA55-WI-034, Revision 0. After closure, the lug gap on the closure ring was 1-1/8" and the ring gap was 9/16". TU-9 was then subjected to a helium leak test. Initially, a  $\Delta P$  of 0.14 kPa was applied which resulted in a leak rate of  $1.0 \times 10^{-6}$  cc He/s thirty-seven seconds into the test. The  $\Delta P$  was then increased to 0.37 kPa for the remainder of the test. This  $\Delta P$  resulted in leak rate of  $4 \times 10^{-6}$  cc He/s two minutes into the test and  $1.0 \times 10^{-5}$  cc He/s after 190 seconds of testing. The leak rate remained relatively stable near this leak rate for the remainder of the leak test with a leak rate of  $1.0 \times 10^{-5}$  cc He/s recorded after 4 minutes of testing and reaching a maximum leak rate of  $2.4 \times 10^{-5}$  cc He/s after 22 minutes of testing. A maximum  $\Delta P$  of 0.37 kPa was used for the tests at the request of the customer due to the likelihood that using the planned  $\Delta P$  of 2 kPa would have resulted in an off scale reading on the helium leak detector (i.e.,  $> 1.0 \times 10^{-4}$  cc He/s).

## 4.3 Analysis of Test Results

The He leak test methodology used in the tests reported on in this document was conceived by LANL and the test apparatus constructed and operated by ORNL specifically for the purpose of testing these containers. This unique test methodology allowed for helium leak tests, which require vacuum conditions, of containers that would have been distorted by being exposed to a pressure differential of approximately 1 atm. This was accomplished by placing the test unit within a chamber, simultaneously pulling a vacuum on both the vacuum chamber and the inside of the test unit, and then, once vacuum conditions were accomplished, isolating the test unit from the vacuum chamber through use of a valve. Another valve was then used to slightly pressurize the test unit with helium thereby creating both a driving force for leakage and a tracer gas to quantitatively measure the leak rate. As the helium leaked from the test unit into the vacuum chamber it was then pulled into the helium leak detector and the leak rate registered and recorded. A suggested revision of the helium leak test apparatus involves the addition of Valve allowing a leak test/QA check of the test manifold prior to test initiation.

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In general, the test units fared poorly when compared to the criteria contained in the Draft DOE Manual 441.1-1. For the design release rate tests, two SNMC's were tested for comparison to design release rate criteria, and one of these units was tested several times. Although TU-1, Test 1, retest 2, TU-1 Test 2, retest 1, TU-1, Test3, retest 1 and TU-5 passed this test, it was not demonstrated that the SNMC design could consistently be assembled and subsequently pass the design release rate criteria. Since containers used in the field cannot be leak tested when in use, it is important that it can be shown that use of a standard assembly procedure consistently results in a high-quality (i.e., leak tight) seal. For the Design Release Rate tests of 5 and 10-gallon UN 1A2 ring-closure drums, it was found that such drums could not meet the leak tight Design Release Leak Rate criteria.

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## 5 DESIGN QUALIFICATION RELEASE RATE TEST

### 5.1 Test Procedure

## 6 The SNMCs and Drums were tested with realistic payloads and under assumed-to-be worst case orientations.

### 6.1

The payload was tungsten shot loaded into a slip lid can. LANL provide three loaded slip lid cans whose various combinations make up the payloads for each container type tested. The slip-lid containers were taped closed, and placed into the SNMC (or Drum), and then assembly procedures were performed on the containers to be tested as stipulated by the test plan.

After assembly, each of the test units was exposed to an immersion bubble leak rate test to ensure that the closure process had resulted in a sealed test unit. The bubble leak rate test consisted of removing the nuclear grade filter on each test unit and attaching a line to the filter opening that allowed air to be pumped into the test unit. After being pressurized with air, each test unit was submerged into a tank of water and the number of bubbles evolved over a 30 second period was counted, and their average size estimated. At the same time, the pressure within the test unit was monitored and recorded at the beginning and at the end of each 30 second test. These test were performed according to TTG Test Procedure TTG-PRF-03, Rev., 0, *Standard Full Boundary Leak Test Method – Immersion Bubble Leak Testing*.

Once the closure of the test unit was verified, each test unit was positioned in the desired drop test orientation on the TTG Small Package Drop Tester and was then raised to the desired drop height over the TTG Indoor Drop Pad. This drop pad consists of a 75.5 in × 48.25 in 2-inch thick steel plate placed atop concrete and re-bar reinforcement. The total mass of the drop pad is over 31,500 lbs. and the qualification of this pad as an essentially unyielding surface is documented in *Design and Certification of Targets for Drop Tests at the NTRC Packaging Research Facility*, ORNL/NTRC-001, Rev. 0. Each test unit was then released from the Small Package Drop Tester and allowed to free fall until impact. Each drop test was videotaped (with the exception of TU-4) and pictures of the damage created by the impact were taken. These test were performed according to TTG Test Procedure TTG-PRF-08, Rev., 1, *Operating Procedure for NCT Drop Testing – Testing of Radioactive Material Packages*.

Drop testing of the test units was intended to challenge the container designs using a *worst-case* impact orientation (i.e., the orientation most likely to cause damage resulting in higher material release rates). However, no analysis or studies were completed prior to

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testing to determine exactly what orientations represented *worst-case* impact damage. In lieu of analysis, experience of the testers (ORNL TTG) in testing small drum-type Type B Shipping Packages was used. This experience suggested that the worst-case orientation would, in general, be a center-of-gravity-over-corner (CGOC) orientation such that the initial impact would be on the lid or closure ring of the container being tested. Therefore, Test Units 1-3 and 5-10 were drop tested in this orientation. Test Unit 4 was dropped in a side orientation because 1) four containers of this type (SNMC, 4-thread) were provided for testing (as opposed to two of each of the other container types) and the protrusion of the handle from the body on the SNMC design suggested a clear weakness in design when considering impact damage effect on material release rate. Had more money, time and test units been available drop test in many other orientations such as end drop on top, end drop on bottom, side drop on container designs not side drop tested, and slap-down (or shallow angle) would have allowed for a more complete gathering of data regarding the ability of these container designs to meet draft Manual requirements.

The CGOC orientation was achieved by finding the point at which the test unit balanced on platen of the Small Package Drop Tester, bracing the package in that position using the bracing arm of the drop tester and then measuring the angle of the package as oriented. It should be noted that even for similar container designs carrying the same payload, the balance points differed due to the fact the payload was free to move inside the container and would be positioned slightly differently from one test to the next. This resulted in significantly different CGOC angles even for test units that were basically identical to one another.

After drop testing was completed, each test unit was again subjected to the immersion bubble leak rate test described above.

## 5.2 Test Results

Copies of the completed check lists and data sheets from these tests are contained in Appendix 3 of this report. Table 6.1 provides the Design Qualification Release Rate test parameters and results. Note that as a primary purpose of the testing was to determine and demonstrate an appropriate test method, some changes were made to the test procedure during the testing. The details of the testing performed on each test unit exposed to drop testing and bubble immersion leak tests are provided below.

After Design Release Rate testing (i.e., helium leak testing) was complete, TU-1 was disassembled and a single slip-lid can with a mass of 12.04 kg was placed within the test unit (Note: TU-1 is described in Section 4.2, above). The empty test unit was found to have a mass of 3.04 kg and the overall assembly had a mass of 15.08 kg. The test unit was then assembled with a closure torque of 140 in-lbs and exposed to an immersion bubble leak rate test. A pressure of 4.8 kPa was applied internally to TU-1 and then TU-1 was submerged under water. There were no bubbles observed during the test.

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## **5.2.1 Test Unit 1 (TU-1) (8-quart, 4-thread SNMC )Test Results**

TU-1 was then oriented at an angle of  $45.3^\circ$ , such that it balanced on the small package drop tester, for a center-of-gravity-over-corner (CGOC) drop test with initial impact on the lid (see Figure 6-1). The unit was then raised to a height of 8 feet above the indoor unyielding surface, in L110 of the National Transportation Research Center, and released. A slight dent at the point of impact on the lid 1.12-inch wide was created and a secondary impact onto the body of TU-1 directly below the point of initial impact created a larger, 1.50-inch wide dent in the body of the TU-1. However, the most notable result of the drop test impact was that the lid of TU-1 slipped a thread approximately  $180^\circ$  from the point of impact. TU-1 was subsequently subjected to post-drop test bubble leakage rate test. During this leak test the unit could not hold pressure and created far too many bubbles to be counted.

## **5.2.1 Test Unit 2 (TU-2) (8-quart, 4-thread SNMC )Test Results**

Test Unit 2 (TU-2) was an 8-quart, 4-thread SNMC serial number 11/05-08043. TU-2 was loaded with a single slip-lid can with a mass of 12.04 kg. TU-2 itself had a mass of 2.99 kg when the nuclear filter was replaced with a leak testing fitting. The overall mass of the assembled TU-2 was 14.99 kg with the nuclear filter in place and 15.03 kg with the leak test fitting in place. The lid of TU-2 was torqued to 140 in-lbs. After initial assembly of TU-2 a water bubble leak test was performed. There was no detected leakage during this test as no bubbles were observed when a pressure of about 4.32 kPa was applied TU-2. TU-2 was then raised to height of 8 feet while positioned in the CGOC orientation ( $44.8^\circ$ ) and dropped onto an unyielding surface (see Figure 6-2). The impact resulted in a minor indentation at the point of impact. After the impact test the test unit was again subjected to a water bubble leak test during which the pressure within TU-2 dropped rapidly and the rate of bubble evolution was far too great to count. The lid of TU-2 was then removed (requiring a torque of 142 in-lbs), the test unit again assembled using a torque of 140 in-lbs, and then again subjected to water bubble leak test. No bubbles were observed during this leak test.

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Table 6.1 Design Qualification Release Rate (Post-Drop) Test Results

Test	Test Unit	Lid Torque (in-lbs)	Mass as Tested (kg)	Drop Height (ft)	Immersion Bubble Leak Testing Results				Meets Criteria/ Comments
					Pre-Drop Test		Post-Drop Test		
					Bubble evolution rate (#/30s)	Pressure drop (kPa)	Bubble evolution rate (#/30s)	Pressure drop (kPa)	
1	TU-1 (8-qt 4- thread SNMC)	140	15.08	8	0	0.01	Too numerous to count	Couldn't hold pressure	Failed
2	TU-2 (8-qt 4- thread SNMC)	140	15.0	8	0	N/R	Too numerous to count	Couldn't hold pressure	Failed
3	TU-3 (8-quart, 4-thread SNMC)	140	15.12	4	0	N/R	Too numerous to count	Couldn't hold pressure	Failed
4	TU-3 (8-quart, 4-thread SNMC)	140	15.12	12	N/P	N/P	N/P	N/P	N/A
5	TU-4 (8-quart, 4-thread SNMC)	140 (increased to 200)	15.05	N/P	Too numerous to count	Couldn't hold pressure	N/P	N/P	Failed prior to drop
6	TU-4 (lid) TU-2 body	140	15.03	8	0	N/R	76	0.60	N/A
7	TU-5 (8-quart, 2- thread SNMC)	140	14.99	8	0	0.12	Too numerous to count	Couldn't hold pressure	Failed
8	TU-6 (8-quart, 2- thread SNMC)	140	15.08	4	0	0.28	Too numerous to count	Couldn't hold pressure	Failed
9	TU-7 5 gallon drum	Bolt Gap 3/8" Ring Gap 3/16"	25.50	8	68	0.26	Too numerous to count	0.66	N/A
10	TU-8 5 gallon drum	Bolt Gap 1/4" Ring Gap 5/16"	25.50	4	13	0.01	~90	0.47	N/A
11	TU-9 10 gallon drum	Bolt Gap 1 1/16" Ring Gap 1/2"	35.0	8	0	0.04	0	0.06	Passed
12	TU-10 10 gallon drum	Bolt Gap 1 1/8" Ring Gap 5/8"	35.0	4	0	0.05	0	0.01	Passed

19209  
10.5 ft  
10.5 ft  
10.5 ft

# DRAFT



**Figure 6-1 Angle measurement prior to TU-1 drop test**



**Figure 6-2 TU-2 on unyielding surface immediately after drop test**

# DRAFT

## 5.2.3 Test Unit 3 (TU-3) (8-quart, 4-thread SNMC )Test Results

Test Unit 3 (TU-3) was an 8-quart, 4-thread SNMC serial number 08/06-08142. TU-3 was loaded with a single slip-lid can with a mass of 12.04 kg. TU-3 itself had a mass of 3.08 kg when the nuclear filter was in place. The overall mass of the assembled test unit TU-3 as drop tested (with the nuclear filter in place) was 15.12 kg. The lid of TU-3 was torqued to 140 in-lbs. After initial assembly of TU-3 a immersion bubble leak test was performed. There was no detected leakage during this test as no bubbles were observed when a pressure of about 5 kPa was applied internally to TU-3. TU-3 was then raised to height of 4 feet while positioned in the CGOC orientation ( $40.0^\circ$ ) and dropped onto an unyielding surface. After the impact test the test unit was again subjected to a water bubble leak test. Initially, the test pressure was set to about 1 kPa and only one bubble was observed. The test pressure was then increased to 5 kPa and the bubble evolution rate increased significantly to the point that the bubbles were too numerous to count and the pressure quickly bled from the test unit. When this test unit was disassembled, a loosening torque of 70 in-lbs was required to loosen the lid.

At the request of LANL, TU-3 was subjected to two additional drop tests not originally included in the test plan. As before, the total mass of the test unit was 15.12 kg including the single 12.04 kg slip-lid can contained inside the test unit. No pre- or post-drop test leakage rate tests were performed during this sequence as the purpose of the drop test was to check for catastrophic failure (i.e., separation of test unit lid from test unit body). The height of the drop test was 12 feet and the test unit was positioned in a CGOC orientation measured at  $39.6^\circ$ . During the free-fall portion of this drop test, the test unit rotated somewhat resulting in a large angle ( $\sim 20^\circ$ ) slap-down impact [i.e., an impact where one end (in this case the lid end) initially impacts the unyielding surface with the test unit at such an angle that an acceleration occurs resulting in a more accelerated impact to the other end of the test unit adjacent to the original point of impact]. While significant denting of the test unit occurred, no catastrophic failure occurred. Because the impact angle was not as originally intended, LANL requested that the drop test be repeated. The test unit was again raised to 12 ft while positioned in a CGOC orientation measured at  $48.2^\circ$  and dropped. For this impact test, the test unit was rotated such that point of impact was  $180^\circ$  from the point of impact of the previous drop test (i.e.  $180^\circ$  from the nuclear filter). On impact, the lid remained on the test unit; however further inspection showed that at about  $180^\circ$  from the impact point the lid had jumped a thread, similar to the result of the 8-ft drop test on TU-1 (see Figure 6-3).

# DRAFT

## 5.2.4 Test Unit 4 (TU-4) (8-quart, 4-thread SNMC )Test Results

Test Unit 4 (TU-4) was an 8-quart, 4-thread SNMC serial number 04/02-08266. TU-4 was loaded with a single slip-lid can with a mass of 12.04 kg. TU-4 itself had a mass of 3.02 kg when the nuclear filter was in place. The overall mass of the assembled test unit TU-4 as drop tested (with the nuclear filter in place) was 15.05 kg. The lid of TU-4 was torqued to 140 in-lbs. After initial assembly of TU-4 the unit was pressurized in preparation for a immersion bubble leak test; however, the test unit would not hold pressure indicating a severe leak. The lid was removed, the O-ring was replaced and the lid was again torque to 140 in-lbs. Again, the test unit would not hold pressure in preparation for the bubble leak test. At this point, the body of TU-2 was retrieved and a new test unit, Test Unit 4/2 (TU-4/2) using the lid of TU-4 and the body of the previously tested TU-2 was assembled using a torque of 140 in-lbs. TU-4/2 was then subjected to an immersion bubble leak test during which there was no detected leakage as no bubbles were observed when a pressure of about 5 kPa was applied internally to TU-4/2. TU-4/2 was then raised to height of 8 feet while positioned in a side-impact orientation in which the primary impact would be directly to the handle that runs along the body, parallel to the axis, of an SMNC and dropped onto an unyielding surface. After the impact test the test unit was again subjected to a water bubble leak test. The test pressure was set to about 5.4 kPa initially, degrading to 4.8 kPa over the 30 second duration of the immersion leak test. Seventy-six bubbles were counted during this 30 second test period. When this test unit was disassembled, a loosening torque of 220 in-lbs was required to loosen the lid.



**Figure 6-3 TU-3 on unyielding surface immediately after final 12 ft drop test with skipped thread visible**

# DRAFT

## 5.2.5 Test Unit 5 (TU-5) (8-quart, 2-thread SNMC )Test Results

After Design Release Rate testing (i.e., helium leak testing) was complete, TU-5 was disassembled, and a single slip-lid can with a mass of 12.04 kg was placed inside (Note: TU-5 is described in Section 4.2, above). The SMNC was found to have a mass of 2.95 kg and the overall mass of TU-5 when reassembled for subsequent testing was 14.99 kg. The closure torque on TU-5 was 140 in-lbs. TU-5 was then subjected to an immersion bubble leak rate test during which no bubbles were observed during a 30 second period in which the pressure inside the TU-5 varied between 5.02 and 4.94 kPa. TU-5 was then raised to height of 8 feet while positioned in the CGOC orientation (53.4°) on the Small Package Drop Tester (see Figure 5-4) and dropped onto an unyielding surface. After the impact test, the test unit was again subjected to an immersion bubble leak test. During the immersion test, the bubbles were too numerous to count and the pressure quickly bled from the test unit. When this test unit was disassembled, a torque of 60 in-lbs was required to loosen the lid.



**Figure 6-4 TU-5 mounted on Small Package Drop Tester prior to drop test (Note: Pink padding on unyielding surface was removed before the drop test was performed.)**

# DRAFT

## 5.2.6 Test Unit 6 (TU-6) (8-quart, 2-thread SNMC )Test Results

Test Unit 6 (TU-6) was an 8-quart, 2-thread SNMC serial number 08/06-08163. TU-6 was loaded with a single slip-lid can with a mass of 12.04 kg. TU-6 itself had a mass of 3.04 kg when the nuclear filter was in place. The overall mass of the assembled test unit TU-6 as drop tested (with the nuclear filter in place) was 15.08 kg. The lid of TU-6 was torqued to 140 in-lbs. After initial assembly of TU-6 an immersion bubble leak test was performed. There was no detected leakage during this test as no bubbles were observed when a pressure of about 5 kPa was applied internally to TU-6. TU-6 was then raised to a height of 4 feet while positioned in the CGOC orientation (45.3°) and dropped onto an unyielding surface. After the impact test the test unit was again subjected to an immersion bubble leak test. During the immersion test, the bubbles were too numerous to count and the pressure quickly bled from the test unit. The loosening torque of this test unit was not recorded.

## 5.2.7 Test Unit 7 (TU-7) (5-gallon drum, Locking Ring) Test Results

After Design Release Rate testing (i.e., helium leak testing) was complete, TU-7 was disassembled and loaded with a single slip-lid can and two stainless steel disks which combined had a mass of 22.07 kg (Note: TU-7 is described in Section 4.2, above). The empty TU-7 was found to have a mass of 3.42 kg and the assembled TU-7 had a mass of 25.50 kg. When initially re-assembled the bolt lugs had a gap of 1/4" and the ring had gap 3/8". TU-7 was subsequently opened and reassembled which resulted in a lug gap of 3/8" and a ring gap of 3/16". TU-7 was then subjected to a pre-drop immersion leak test during which 68 bubbles with an approximate diameter of 7 mm were counted over a 30 second period. During this leak test the pressure inside TU-7 varied between 5.62 (at the beginning) and 5.36 (at the end) kPa. TU-7 was then raised to height of 8 feet while positioned in the CGOC orientation (51.3°) such that initial impact would be on the closure lug for the planned drop test onto the unyielding surface. However, prior to being intentionally released, but after reaching the full intended drop height of 8 feet, TU-7 fell off of the precision drop tester platform. The package rotated a full 360° during the unintended drop and landed directly on the closure lug as intended; however, the impact angle may have been considerably different than planned. Because the drop happened before it was intended to, the video camera was not operating such that there is no way to review the actual impact to determine the angle at the time of impact. As a result of the impact, the lid of TU-7 was significantly deformed (see Figure 6-5). After the impact test the test unit was again subjected to an immersion bubble leak test. During the 30 second immersion test, the bubbles were too numerous to count and the pressure within the vessel dropped from 4.80 (at the beginning) to 4.14 (at the end) kPa.



**Figure 6-5 TU-7 lid deformation near closure ring bolt lugs after drop testing**

## **5.2.8 Test Unit 8 (TU-8) (5-gallon drum, Locking Ring) Test Results**

Test Unit 8 (TU-8) was a stainless steel, standard 5-gallon drum and lid with locking-ring closure, serial number 10/02-5099 which had been fitted with a filter port in the lid. Integral to the lid was a black elastomeric gasket positioned to create a seal between the lid and drum body when assembled. TU-8 was loaded with a single slip-lid can and two stainless steel disks which combined had a mass of 22.07 kg. The empty TU-8 was found to have a mass of 3.43 kg and the assembled TU-8 had a mass of 25.50 kg. TU-8 was assembled according to LANL TA-55 Work Instruction TA55-WI-034, Revision 0. After closure, the lug gap on the closure ring was 1/4" and the ring gap was 5/16". TU-8 was then subjected to a pre-drop immersion leak test during which 13 bubbles of an unspecified diameter were counted over a 30 second period. During this leak test the pressure inside the TU-8 varied between 4.34 (at the beginning) and 4.33 (at the end) kPa. TU-8 was then raised to height of 4 feet while positioned in the CGOC orientation (54.7°) with the closure lug targeted for initial impact and dropped onto the unyielding surface. After the impact test the test unit was again subjected to an immersion bubble leak test. During the 30 second immersion test, approximately 90 bubbles (very difficult to count at the rate evolved) of an estimated diameter of 3 mm were observed and the pressure within the vessel dropped from 4.82 (at the beginning) to 4.35 (at the end) kPa.

# DRAFT

## 5.2.9 Test Unit 9 (TU-8) (10-gallon drum, Locking Ring) Test Results

After Design Release Rate testing (i.e., helium leak testing) was complete, TU-9 was disassembled and loaded with a two slip-lid cans and a single stainless steel disk which combined had a mass of 28.18 kg (Note: TU-9 is described in Section 4.2, above). The empty TU-9 was found to have a mass of 6.82 kg and the assembled TU-9 had a mass of 35.0 kg. When re-assembled the bolt lugs had a gap of 1-1/16" and the ring had a 1/2" gap. TU-9 was then subjected to a pre-drop immersion leak test during which no bubbles were observed over a 30 second period. During this leak test the pressure inside the TU-9 varied between 5.81 (at the beginning) and 5.77 (at the end) kPa. TU-9 was then raised to height of 8 feet while positioned in the CGOC orientation (51.6°) such that initial impact onto the unyielding surface would be on the closure lug and dropped. After the impact test, the test unit was again subjected to an immersion bubble leak test. During the 30 second immersion test, no bubbles were observed. During this leak test the pressure inside the TU-9 varied between 4.68 (at the beginning) and 4.62 (at the end) kPa.



**Figure 6-6 TU-9 lid deformation near closure ring bolt lugs after drop testing**

# DRAFT

## 5.2.10 Test Unit 10 (TU-10) (10-gallon drum, Locking Ring) Test Results

Test unit 10 (TU-10) was a stainless steel, standard 10-gallon drum and lid with locking-ring closure, serial number 6/02-10043 which had been fitted with a filter port in the lid. Integral to the lid was a black elastomeric gasket positioned to create a seal between the lid and drum body when assembled. TU-10 was loaded with two slip-lid cans, a single stainless steel disk, and a spare bolt (taped to the lid of one of slip-lid cans) which combined together had a mass of 28.22 kg. The empty TU-10 was found to have a mass of 6.80 kg and the assembled TU-10 had a mass of 35.0 kg. TU-10 was assembled according to LANL TA-55 Work Instruction TA55-WI-034, Revision 0. After closure, the lug gap on the closure ring was 1-1/8" and the ring gap was 5/8". TU-10 was then subjected to a pre-drop immersion leak test during which no bubbles were observed during a 30 second period. During this leak test the pressure inside the TU-10 varied between 5.53 (at the beginning) and 5.48 (at the end) kPa. TU-10 was then raised to height of 4 feet while positioned in the CGOC orientation (56.4°) with the closure lug targeted for initial impact and dropped onto the unyielding surface. After the impact test, the test unit was again subjected to an immersion bubble leak test. During the 30 second immersion test, no bubbles were observed. During this leak test the pressure inside the TU-10 varied between 5.46 (at the beginning) and 5.45 (at the end) kPa.

# DRAFT

## 7 SUMMARY

### 7.1 Testing Methods

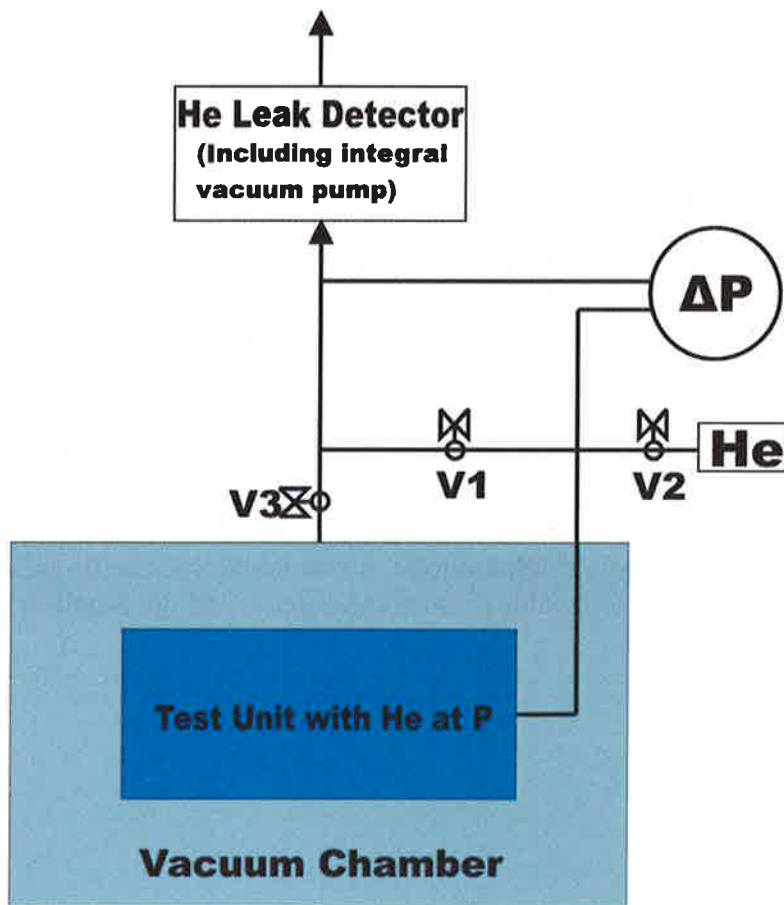
The test methodology and leak test apparatus worked very well. The pressure differential was kept low enough to ensure that test unit distortion was not an issue. Leak rates were successfully measured and subsequently translated into equivalent air leakage rates under standardized or “reference” conditions.

It is recommended that a third valve be introduced into the test apparatus (See **Figure 7-1**) such that a simple test of the vast majority of the test manifold can be quickly performed prior to test initiation to ensure that that portion of the leak test apparatus is assembled correctly (i.e., leak tight). Additionally, it is recommended that a dedicated procedure, specifically for use with this test apparatus, be developed rather than using a generic helium leak test procedure developed for leak testing of Type B shipping packages.

The immersion bubble leak rate procedure used in the tests reported on in this document was developed specifically for these tests. The procedure requires that the average diameter of the evolving bubbles be estimated in order to determine a leak rate. In practice, it is very difficult to accurately estimate the average size as often times bubbles of various sizes are being produced. However, the rate at which the pressurization within the test units decayed during a specified time period was recorded. This data (pressure decay rate) can be used to calculate a leak rate. It appears that pressure decay is likely a more accurate method of determining leak rate than bubble counting.

Drop tests were performed using the TTG Small Package Drop Tester. The drop test device itself worked very well and does not impart any spin to the test unit when released. The test unit’s impact orientation is equal to the rigged orientation. However, there was one drop test in which the test unit released earlier than planned. The methodology through which the test unit is held in place on the Small Package Drop Tester should be inspected and repaired as necessary or individual rigs should be manufactured for each type of test unit to be tested such the overhead crane can be used instead of the Small Package Drop Tester for drop test release.

# DRAFT



**Figure 7-1** Suggested revision of the helium leak test apparatus, with the addition of Valve 3, allowing a leak test/QA check of the test manifold prior to test initiation

# DRAFT

## 7.2 Container Test Results

In general the test units fared poorly when compared to the criteria contained in the Draft DOE Manual 441.1-1. The following observations are provided:

- For the design release rate tests, two SNMC's were tested for comparison to design release rate criteria, and one of these units was tested several times. Although TU-1, Test 1, retest 2, TU-1 Test 2, retest 1, TU-1, Test3, retest 1 and TU-5 passed this test, it was not demonstrated that the SNMC design could consistently be assembled and subsequently pass the design release rate criteria. Since containers used in the field cannot be leak tested when in use, it is important that it can be shown that use of a standard assembly procedure consistently results in a high-quality (i.e., leak tight) seal.
- For the Design Release Rate tests of 5 and 10-gallon UN 1A2 ring-closure drums, it was found that such drums could not meet the leak tight Design Release Leak Rate criteria.
- For the Design Qualification Release Rate, it was found that the SNMC designs, after drop testing from either 8 feet or 4 feet could not meet the Design Qualification Release Rate criteria.
- For the Design Qualification Release Rate, it was found that the 10-gallon UN 1A2 ring-closure drums appeared to be able to meet the criteria, and the 5-gallon UN 1A2 drum probably met the criteria.

# DRAFT

## Appendix 1 - Container Design Details

Both the 4-thread and the 2-thread LANL SNMC containers consisted of a body with an approximate diameter of 8.5 in and 11.0 in tall (See Figure A-1 below). On the outside of the body, at the top, were threads used in the attachment of the lids. An elastomeric O-ring sat in a groove above the threads such that when a lid was attached the O-ring was to form a seal with the flat portion of the lid (See Figure A-2 below).



Figure A-1 TU-1 (8-quart, 4-thread, SNMC) prior to testing

# DRAFT



**Figure A-2 TU-2 (8-quart, 4-thread, SNMC) O-ring and thread detail**

Figure A-3 shows a comparison of the threads in the lid of a 4-thread test unit (TU-1 through TU-4) and a 2-thread test unit (TU-5 and TU-6). Note that the all SNMC test units had the same thread on the test unit body; however, Test Units 1 through 4 had four threads in the lid whereas Test Units 5 and 6 had only 2 threads in the lid.



**Figure A-3 Comparison of lids of a 4-thread SNMC (left) and a 2-thread SNMC (right)**

# DRAFT

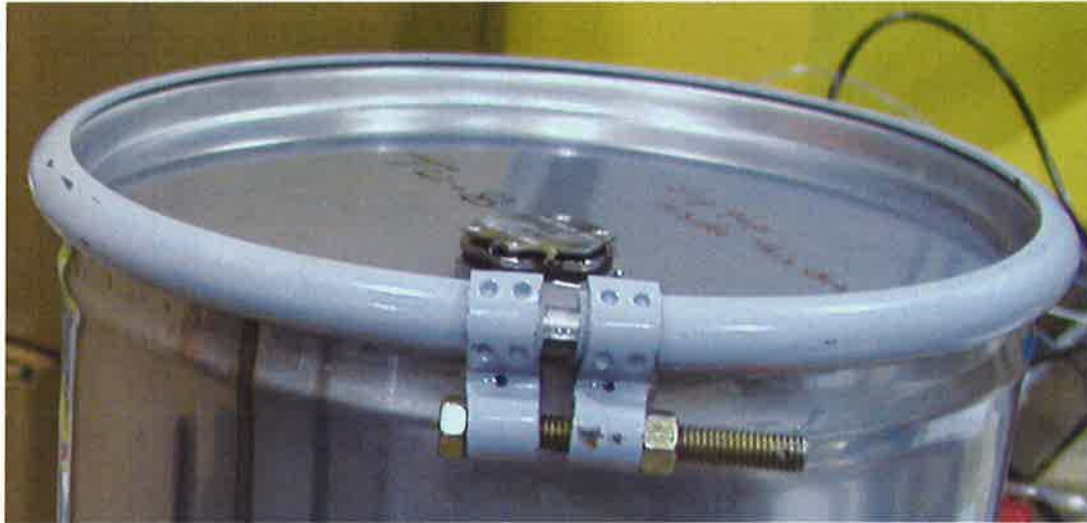
The 5-gallon ring-closure drums utilized for TU-7 and TU-8 were constructed of stainless steel and carried UN 1A2 markings. These test units were approximately 13.25 in tall and 12 in diameter (See Figure A-4 below).



**Figure A-4 TU-8 (5-gallon, stainless steel, ring-closure drum) prior to testing**

# DRAFT

Closure of these tests units was accomplished by a standard ring closure with a single bolt (See Figure A-5 below). A black elastomeric gasket was part of each test unit's lid and was wedged between the lid and the drum body during the closure process.



**Figure A-5 Close up of TU-8 (5-gallon, stainless steel, ring-closure drum) ring closure**

The 10-gallon ring-closure drums utilized for Test units 9 and 10 were constructed of stainless steel and carried UN 1A2 markings. These test units were approximately 18.75 in tall and 14.75 in diameter (See Figure A-6 below). Closure of these tests units was accomplished by a standard ring closure with a single bolt. A black elastomeric gasket was part of each test unit's lid and was wedged between the lid and the drum body during the closure process.

# DRAFT

All ten test units were fitted with nuclear grade filters in the lid. The filters are attached through threaded openings in the container lids. The design and size of all of the filters was the same (a typical filter can be seen on the lid of TU-9 in Figure A-6, below). The filters were left in place for drop test activities. The filters were removed and the threaded openings used to mate the test units to the leak test apparatus for all helium and immersion bubble leak tests.



**Figure A-6 TU-10 (10-gallon, stainless steel, ring-closure drum)**

# **DRAFT**

## **Appendix 2 - Test Procedures**

**Provide an example of the test procedures used to support this testing**

# DRAFT

## Appendix 3 - Checklists and Data Sheets from All Tests

**Provide an example of the checklist and data sheet s used to support this testing**

# DRAFT

**Appendix 4 (included instead, by reference in the body of the report, to the published calculation document already publically available)**

## **DETAILS OF CALCULATION OF DESIGN RELEASE RATE AND DESIGN QUALIFICATION RELEASE RATE**

The following outlines the calculation of design release rate and design qualification release rate for the testing process utilized. The calculation is for weapons grade Pu in an oxide form stored in a vented container. This calculation was performed in accordance with the ANSI N14.5-1997 methodology.

### **Calculation Overview**

- Step 1: Determine the mass release rate criterion (i.e., convert  $10^{-6} A_2/\text{hr}$  to g/hr)
- Step 2: Convert the mass release rate criterion to a volumetric release rate criterion (air and He gas)
- Step 3: Determine driving pressure for release of material specific to the container and facility conditions
- Step 4: Convert the He gas release rate criterion (for the facility conditions) to a test acceptance criteria specific to the He gas detector being utilized for the test.
- Step 5: Calculate a post-drop test release rate (corresponding to  $10^{-3} A_2/\text{event}$ )

### **Step 1: Determine the mass release rate criterion**

The design release rate criterion is  $10^{-6} A_2$  per hour.

Using the value for  $A_2$  (.352 g) for weapons grade Pu oxide (taken from Table 3 in Attachment 3 of this Manual) this corresponds to:

$$\begin{aligned} 10^{-6} A_2/\text{hr} &= 10^{-6} (0.352 \text{ g})/\text{hr} = 3.52 \times 10^{-7} \text{ g/hr} (1 \text{ hr}/3600 \text{ s}) \\ &= 9.8 \times 10^{-11} \text{ g/s} \end{aligned}$$

# DRAFT

## **Step 2: Convert to a volumetric release rate Criterion**

In order to convert the mass release rate into a volumetric release rate, the density of the Pu oxide must be determined.

The oxide aerosol density used by ANSI N 14.5-1997 (Section B.15.27) is  $9 \times 10^{-6} \text{ g/cm}^3$  and represents the worst case of an agitated container intended to simulate vibrations present in normal transport. This is more conservative than the case for a quiescent package in storage or carefully handled by plutonium workers. An extensive literature review of experimental studies of aerosol actinide oxide densities achieved by flowing gas through a vibrating bed is summarized in NUREG/CR-6487, *Containment Analysis for Type B Packages used to Transport Various Contents*. The lines of best fit yielded densities of  $1 \times 10^{-8} \text{ g/cm}^3$  for a leak above the oxide bed and  $6 \times 10^{-8} \text{ g/cm}^3$  for a leak below the oxide bed.

For conservatism and simplicity this oxide aerosol density is rounded up to  $1 \times 10^{-7} \text{ g/cm}^3$  which is used as the oxide density in the fluid leaking from the container in this calculation.

Therefore, the volumetric gas (air) release rate at the pressure and temperature of the container is:

$$\frac{\text{Pu Mass Release Rate}}{\text{Pu Gas Concentration}} = \frac{9.8 \times 10^{-11} \text{ g/s}}{1 \times 10^{-7} \text{ g/cm}^3} = 9.8 \times 10^{-4} \text{ cm}^3/\text{s} \text{ for Weapons Grade Pu oxide}$$

## **Step 3: Determine driving pressure for release of material**

The driving pressure from the containers is a combination of pressure buildup in the container from radiolytic gas generation and changes in atmospheric pressure conditions. For this example, a filtered container is used.

Volume of gas generated by radiolysis:

$$\Delta V_g = \frac{G f_{\text{H}_2\text{O}} \text{TA} \Delta t}{P}$$

Where  $G$  = gas generation rate (from the DNFSB 94-1 Surveillance and Monitoring Program<sup>2</sup>) is equal to 300 nmol/s Watt for Pu.

$f_{\text{H}_2\text{O}}$  = fraction of water in contained material (.05 is assumed for this example)

$\text{TA}$  = Total Activity of contained material in Watts (determined from total material in the container. For this example 3.78 Watts is used which is reasonable for a one quart container loaded with Pu)

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<sup>2</sup>LA-13261-PR, 94-1 Research and Development Project, December 1996

# DRAFT

R = Gas constant (8.314 kPa L/K mol)

T = Local temperature (assumed to be 55 °C [328 °K])

P = Local atmospheric pressure (assumed to be 100 kPa)

$$\Delta V_g = \frac{\left(300 \times 10^{-9} \frac{\text{mol}}{\text{s W}}\right)(0.05)(3.78 \text{ W})\left(8.31 \frac{\text{kPa L}}{\text{K mol}}\right)(328 \text{ K})}{100 \text{ kPa}} = 1.55 \times 10^{-6} \text{ liter/s}$$

$$\left(1.55 \times 10^{-6} \text{ liter/s}\right)\left(1000 \frac{\text{cm}^3}{\text{liter}}\right) = 1.55 \times 10^{-3} \text{ cm}^3/\text{s}$$

This change in volume is converted to a pressure drop across the filter utilizing data on the filter flow capacity (for this case 200 ml/min at 1 inch water column differential pressure [.248 kPa]) assuming that the filter is 99 percent clogged. This flow per pressure drop is then:

$$F = \frac{(200 \text{ ml/min})(1 \text{ cm}^3/\text{ml})(1 \text{ min}/60 \text{ s})}{(0.248 \text{ kPa})} = 13 \frac{\text{cm}^3}{\text{s kPa}}$$

For a 99 percent clogged filter the flow is  $F_{\text{red}} = F * 10^{-2}$

$$\Delta P_g = \frac{\Delta V_g}{F_{\text{red}}} = \frac{1.55 \times 10^{-3} \frac{\text{cm}^3}{\text{s}}}{\left(13 \frac{\text{cm}^3}{\text{s kPa}}\right)(10^{-2})} = 0.012 \text{ kPa}$$

The driving pressure due to atmospheric pressure changes can be determined from records of maximum daily atmospheric pressure changes. For example, the maximum atmospheric pressure change in a day at LANL was 2 kPa. This pressure change could be directly used as the driving pressure for the release of material. Conversely a calculation of the volume that must be released through a filter vent can be determined and an assumption made on the time the pressure change occurs.

For example (using the ideal gas law for the case of a 1 quart [.95 liter] container), assuming the pressure change occurred over a 2 hour time, the change in volume due to atmospheric pressure changes is:

$$8 \Delta V_a = \frac{\Delta P}{P} \cdot V = \frac{2 \text{ kPa}}{100 \text{ kPa}} \cdot \frac{1}{2 \text{ hr}} \cdot (.95 \text{ liter}) \left( \frac{1000 \text{ cm}^3}{\text{liter}} \right) \left( \frac{1 \text{ hr}}{3600 \text{ s}} \right) = 2.6 \times 10^{-3} \frac{\text{cm}^3}{\text{s}}$$

# DRAFT

This change in volume can be converted into a change in differential pressure from the atmospheric pressure change:

$$\Delta P_a = \frac{\Delta V_a}{F_{red}} = \frac{2.6 \times 10^{-3} \frac{\text{cm}^3}{\text{s}}}{\left(13 \frac{\text{cm}^3}{\text{s kPa}}\right) (10^{-2})} = 0.02 \text{ kPa}$$

$$\Delta P_{total} = \Delta P_a + \Delta P_g = 0.02 \text{ kPa} + 0.012 \text{ kPa} = 0.032 \text{ kPa}$$

Therefore, for a 1 liter container, an appropriate differential pressure to conduct a leak rate test would be a 0.032 kPa differential pressure. For larger containers with more material using the same filter,

the differential pressure would increase to 1 to 2 kPa. For most storage conditions, a 2 kPa differential pressure would be conservative.

## **Step 4: Convert the He gas release rate criterion to a test acceptance criterion**

A means for performing the release rate test is to pull a vacuum (exhausting to a He detector) on the outside of the container and introduce He into the container such that there is a 1 kPa differential pressure.

However, because the He leak detector measures the release rate in standard [std] atmosphere [atm]  $\text{cm}^3 \text{sec}^{-1}$  ( $= 101 \text{ kPa cm}^3 \text{sec}^{-1}$ ), a conversion must be made to account for the density differences between the vacuum and the standard atmosphere. Using the approach specified in ANSI N 14.5, the gas leak rate can be converted to a He leak rate at the maximum operating differential pressure. Using 1 kPa differential pressure for a container with Weapons Grade Pu oxide, the volumetric gas release rate determined in Step 2 can be converted to a He leak rate reported by a He leak detector in  $\text{atm cm}^3/\text{s}$  of He.

Several steps are involved in performing this calculation. First, the size of the leakage hole needs to be determined that corresponds to the air leakage rate (and conditions), then a He leak rate corresponding to testing conditions and the calculated leakage hole size.

The following conditions are used for the calculation of the hole size:

Pressure upstream =  $P_u = 102 \text{ kPa} = 1.01 \text{ atm}$

Pressure downstream =  $P_d = 101 \text{ kPa} = 1.00 \text{ atm}$

Average pressure =  $P_a = \frac{1}{2}(P_u + P_d) = \frac{1}{2}(1.01 + 1.00) = 1.005 \text{ atm}$

Measured Leak Rate =  $L_u = 9.8 \times 10^{-4} \text{ cm}^3/\text{sec}$

Temperature =  $T = 298\text{K}$

Gas = air

Gas Properties:

Fluid Viscosity:  $\mu = 0.0185 \text{ cP}$

Molecular Weight:  $M = 29 \text{ g/gmol}$

Length of leakage pathway (assumption) =  $0.1 \text{ cm}$

# DRAFT

**Calculate the hole diameter using ANSI N14.5 Equation B.3, B.4 and B.5 using the approach outlined in example B5:**

Calculate  $F_c$  (coefficient of continuum flow conductance, ANSI N14.5 Eq B.3):

$$F_c = \frac{\left[ 2.49 \times 10^6 D^4 \right] \frac{cm^3}{atm \cdot s}}{a\mu} = \frac{\left[ 2.49 \times 10^6 D^4 \right] \frac{cm^3}{atm \cdot s}}{(0.1)(0.0185)} = 1.35 \times 10^9 D^4 \frac{cm^3}{atm \cdot s}$$

Calculate  $F_m$  (coefficient of free molecular flow ANSI N14.5 Equation B.4):

$$F_m = \frac{\left[ 3.81 \times 10^3 D^3 \left( \frac{T}{M} \right)^{0.5} \right] \frac{cm^3}{atm \cdot s}}{aP_a} = \frac{\left[ 3.81 \times 10^3 D^3 \left( \frac{298}{29} \right)^{0.5} \right] \frac{cm^3}{atm \cdot s}}{(0.1)(1.005)} = 1.21 \times 10^5 D^3 \frac{cm^3}{atm \cdot s}$$

Putting this information into ANSI N 14.5 Equation B.5 (where  $L_u$  is the upstream air leakage rate)

$$L_u = (F_c + F_m)(P_u - P_d) \left( \frac{P_a}{P_u} \right) \frac{cm^3}{s}$$
$$9.8 \times 10^{-4} \frac{cm^3}{s} = \left( 1.35 \times 10^9 D^4 \frac{cm^3}{atm \cdot s} + 1.21 \times 10^5 D^3 \frac{cm^3}{atm \cdot s} \right) (1.01 atm - 1.00 atm) \left( \frac{1.005 atm}{1.01 atm} \right)$$

Solve for D in order to obtain:  $D = 2.9 \times 10^{-3} cm$ .

**Calculate the helium leak rate at the testing conditions:**

The following conditions apply to this situation

Pressure upstream =  $P_u = 1 \text{ kPa}$  ( $1 \text{ atm}/101 \text{ kPa}$ ) =  $0.01 \text{ atm}$

Pressure downstream =  $P_d = 0 \text{ atm}$

Temperature =  $T = 298K$

$D = 2.9 \times 10^{-3} \text{ cm}$

Gas = Helium

# DRAFT

Gas Properties:

$$\mu = 0.0198 \text{ cp}$$

$$M = 4 \text{ g/gmol}$$

Calculate the leak rate:

$$P_a = \frac{1}{2}(P_u + P_d) = \frac{1}{2}(0.01 + 0) = .005 \text{ atm}$$

Calculate  $F_c$  (ANSI N14.5 Eq B.3):

$$F_c = \frac{\left[ 2.49 \times 10^6 D^4 \right] \frac{\text{cm}^3}{\text{atm s}}}{a\mu} = \frac{\left[ (2.49 \times 10^6) (2.9 \times 10^{-3})^4 \right] \frac{\text{cm}^3}{\text{atm s}}}{(1)(0.0198)} = 0.088 \frac{\text{cm}^3}{\text{atm s}}$$

Calculate  $F_m$  (ANSI N14.5 Equation B.4):

$$F_m = \frac{\left[ 3.81 \times 10^3 D^3 \left( \frac{T}{M} \right)^{0.5} \right] \frac{\text{cm}^3}{\text{atm s}}}{aP_a} = \frac{\left[ (3.81 \times 10^3) (2.9 \times 10^{-3})^3 \left( \frac{298}{4} \right)^{0.5} \right] \frac{\text{cm}^3}{\text{atm s}}}{(0.1)(0.005 \text{ atm})}$$
$$= 1.6 \frac{\text{cm}^3}{\text{atm s}}$$

Putting this information into ANSI N 14.5 Equation B.5 (where  $L_u$  is the helium leak rate and  $Q$  is the mass flow rate detected by the He leak detector)

$$L_u = (F_c + F_m)(P_u - P_d) \left( \frac{P_a}{P_u} \right) \frac{\text{cm}^3}{s}$$

$$L_u = \left( 0.088 \frac{\text{cm}^3}{\text{atm s}} + 1.6 \frac{\text{cm}^3}{\text{atm s}} \right) (0.01 \text{ atm} - 0.00 \text{ atm}) \left( \frac{.005 \text{ atm}}{.01 \text{ atm}} \right) = 8.4 \times 10^{-3} \frac{\text{cm}^3}{s}$$

$$Q = L_u \times P_u = 8.4 \times 10^{-3} \frac{\text{cm}^3}{s} \times 0.01 \text{ atm} = 8.4 \times 10^{-5} \frac{\text{atm cm}^3}{s}$$

The Helium Leak Rate Test Criteria is calculated to be  $8.4 \times 10^{-5} \text{ atm cm}^3/\text{s}$ .

Note: Because the He detector is not being operated over a 1 atm pressure differential, the actual reading will be different than the leak rate. This is calculated by and adjustment using the upstream pressure.

# DRAFT

## **Step 5: Establish a post-drop release test criterion**

The design qualification release rate criterion ( $10^{-3}A_2$  per event) can be measured as either as a material release amount after the drop (in a given period of time, e.g., 10 minutes) or a gas release rate after the drop. The measurement of material provides a better indication of the potential impact on a worker (because it accounts for the potential puff release that may occur) but may be impracticable because the release criterion is extremely small for certain high activity isotopes (e.g., .00035 grams of weapons grade Pu).

A simple go/no go test can be used on material that has an extremely small release criterion by loading the container with a luminescent surrogate material (e.g., fluorescein) and then looking for any release of material during and immediately following the drop test.

Alternatively, since a zero release criteria may be too restrictive, if very small amounts of surrogate material are released, an assessment of the post drop release rate can serve as an appropriate indicator of the ability of the container to meet the post drop release rate criterion.

Using the methods outlined in ANSI N 14.5-1997, for Weapons Grade Pu, a bubble test (with a criterion of no bubbles released) or a pressure drop test, may be shown to suffice.

## Testing and Validation of Threaded Lid Vented Nuclear Materials Storage Containers

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 (2) Safe Sites Of Colorado, RFETS, Box 464, Golden, CO 80403

A type 304 stainless steel nuclear materials storage container (NMC) has been developed and tested to verify leak tightness under severe impact. The nuclear materials containers were designed to meet requirements established in DOE's Recommendation 94-1, "Criteria For Interim Safe Storage of Plutonium-Bearing Solid Material". Containers used for the interim storage (less than 20 years) of nuclear materials must allow a means for retrieval and observation of stored contents, while also meeting stringent structural and leak-proof requirements. The new, all stainless steel container has a threaded lid closure that is equipped with a sintered stainless steel high efficiency particulate air (HEPA) filter vent. The container, which can be hand tightened, provides a positive seal for storing nuclear material, yet allows the controlled release of explosive gases including hydrogen.

The nuclear materials container, which was designed for easy retrieval and monitoring, features a lid which can be tightened and released by hand via a handle. To allow the controlled release of gas and simultaneous retention of radioactive particulate, a type 316 stainless steel HEPA filter is welded to the interior surface of the lid. A semi permeable membrane is adhered to the top surface of the container to prevent water entry up to 2 PSI. A handle allows retrieval and lid installation. Figure -1 shows a photograph the nuclear material storage container.

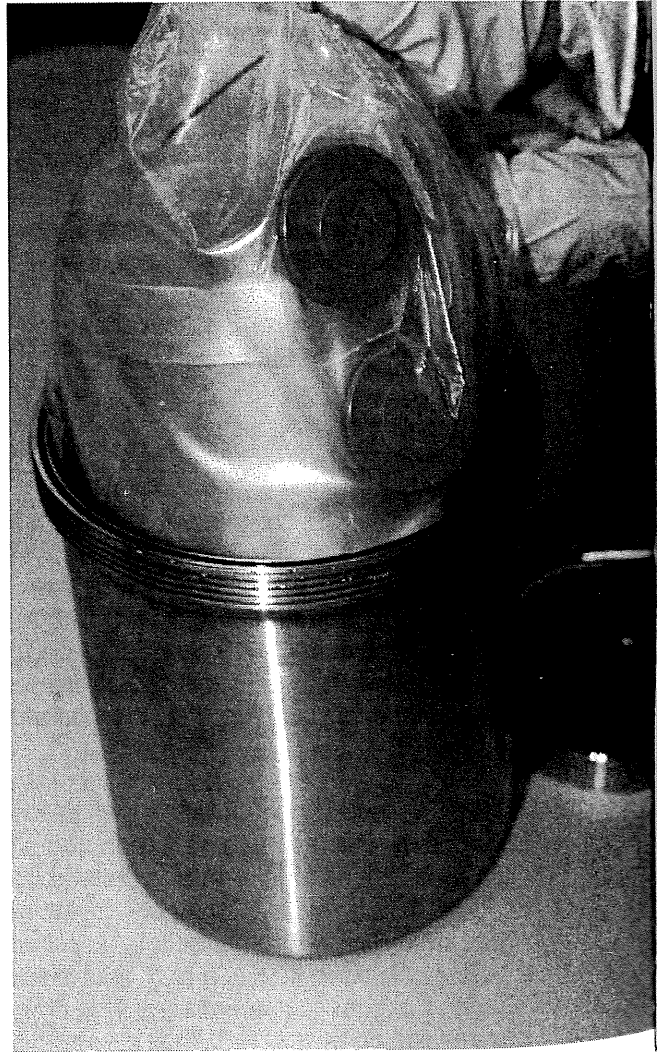
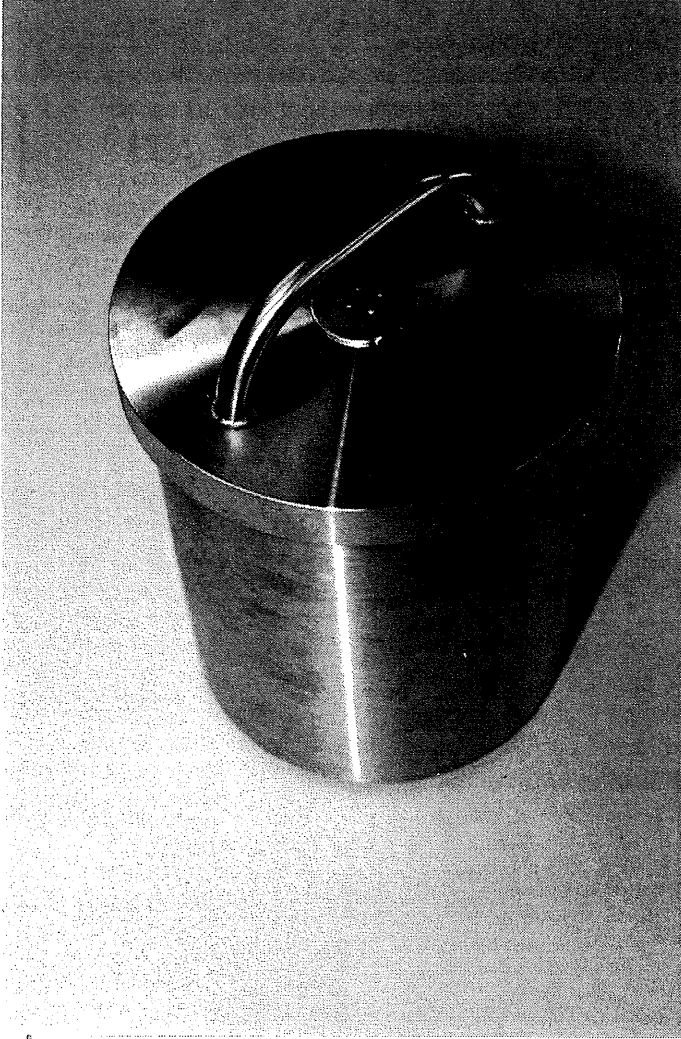
Results of air leak tests after a 1.21 meter drop test, hydrogen diffusivity measurements, particle retention and gas permeability, and water intrusion tests on the nuclear material containers (NMC) will be presented.

### DROP TESTS

One test criterion for the container is that, with its packaged contents, it must remain leak tight after a 1.21 meter drop onto an un-yielding surface. The containers were dropped from the specified height in five orientations; flat on the bottom, 45 degrees off of normal on the bottom corner, directly on the handle, direct side impact, and at roughly 45 degrees off of normal on the screw-top. Observations were made of any visible changes to container integrity or appearance. Also, when possible, the exact angle of impact was measured and recorded. Leak tests were conducted by observing the inverted container, immersed in water, for bubbles after the container had been internally pressurized to a minimum of 5" water column.

The package in the test configuration was as nearly duplicated to an actual package as possible. A pewter container, which serves as a radiation shield for 60 Kev gamma radiation, was filled with about 3.6 kilograms of dry sand to simulate the stabilized nuclear material. The pewter can was double bagged; the first bag was a 12 mil polyvinylchloride (PVC) bag with a NucFil®-030 (patented) snap-fit HEPA filter affixed to it, and the second layer was a 3 mil polyethylene bag, also with a NucFil-030 snap-fit HEPA filter affixed to it. Each bag was thermally heat sealed and then trapped air evacuated. The double bagged pewter can full of sand was then inserted into the threaded nuclear material storage container and the screw top threaded on and hand tightened. Figure-2 shows a photograph of the double bagged lead free pewter can being installed into the nuclear material storage container.

The required drop test height is from greater than 1.21 meters onto an unyielding surface. Each container was dropped from about 1.21 meters by hand which allowed the best control for providing impact with the cement floor in the desired orientation.



**Figure -2 Doubled bagged lead free pewter can**

## LEAK RATE TESTS

The leak rate test was done by internally pressurizing the container to about 5" water column pressure differential. Then the container was immersed upside down in a basin of water. The water temperature was maintained at nearly room temperature (22 degrees centigrade). Figure - 3 shows a photograph of two of the pressurized containers before immersion into the water basin. Figure -4 shows a photograph the two containers immersed in the basin of water with the pressure gage reading slightly above 5 inches water column. Upon the initial immersion, air bubbles escape out from the threads for about 30 seconds. The container was rotated by hand while tapping on the lid to encourage the trapped air in the threads to escape, prior to monitoring for container leakage. Leaks are detected quite easily if they exist because air bubbling continues. A passing result is one where there are no bubbles detected for more than three minutes. A continuous stream of a air bubbles would be grounds for failing the container.

## DROP TEST RESULTS

Figure-1 shows a photograph of the canister prior to being dropped from the test height.. Figure 5 shows a photograph of a test container just prior to drop test from a height of about 1.21 meters. The results of leak rate testing after drop tests in five orientations are given in TABLE -1.

**TABLE - 1 RESULTS OF 1.21 (4 Feet) METER DROP TEST**

<b>DROP TEST ORIENTATION</b>	<b>CAN #</b>	<b>OBSERVATIONS NOTED</b>	<b>LEAK RATE TEST PASS / FAIL</b>
DIRECT ON BOTTOM	RFP-2	SLIGHT INDENTATION AT POINT OF IMPACT 10 mm long, 1 mm	PASS
BOTTOM EDGE	RFP-2	IMPACTED AT 22 DEGREES SOME INDENTATION 41 mm long, 5.61 mm deep	PASS
DIRECT ON HANDLE	RFP-3	FLATTENED TOP EDGE OF HANDLE SLIGHTLY 1mm, LID REMAINS FLAT	PASS
DIRECT ON EDGE OF LID	RFP-3	ONLY SLIGHT BLEMISH AT POINT OF IMPACT.	PASS
DIRECT ON SIDE	RFP-4	SLIGHT INDENTATION ON BOTTOM END, SLIGHT BLEMISH ON EDGE OF LID	PASS

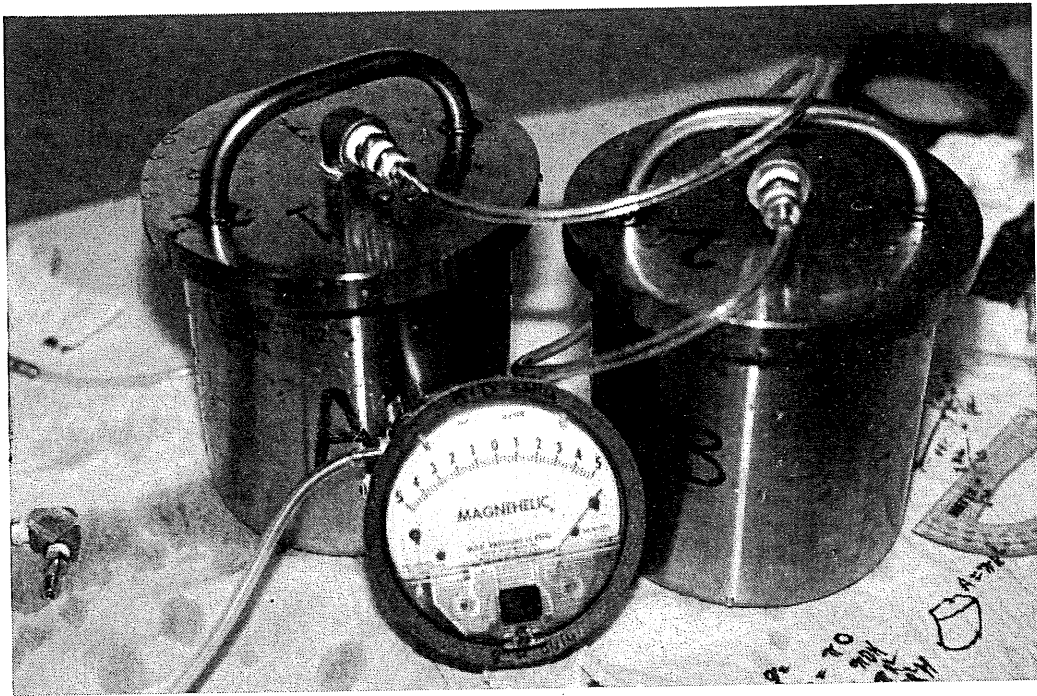


Figure -4 Pressurized Leak Test

After successfully passing the required drop test from 1.21 meters, the containers were dropped in the same orientations from 2.5 meters (8 feet). The drop tests were conducted at NFT Inc. Laboratory, Golden, CO USA and data recorded in laboratory notebook -01 pages 56 through 58. The tests were also videotaped and witness by an independent party. The results are shown in Table -2 below:

**TABLE-2 RESULTS OF 2.5 (8 feet) METER DROP TEST**

<u>ORIENTATION</u>	<u>CAN #</u>	<u>OBSERVATIONS</u> <u>NOTED</u>	<u>LEAK RATE</u> <u>TEST PASS / FAIL</u>
DIRECT BOTTOM EDGE	RFP-4	IMPACT AT 34 DEGREES OFF NORMAL, SEVERE WRINKLES ON LOWER EDGE, 88 mm long, 12.4 mm DEEP	PASS
DIRECT ON HANDLE	RFP-4	INDENTED HANDLE 2.44mm	PASS
DIRECT ON EDGE OF LID	RFP-4	MARRED SURFACE FINISH NO DENTS	PASS

#### HYDROGEN DIFFUSIVITY

A hydrogen diffusivity test was conducted to verify that gases generated would diffuse out of the container through the filter and membrane. The hydrogen diffusivity test quantifies the rate at which radiolytically and chemically generated hydrogen and other gases will diffuse with zero pressure gradient across the membrane. The Waste Isolation Pilot Plant Safety Analysis Report section 1.3.5 requires a value of  $1.9 \text{ E-06}$  mole/second/mole fraction. A gas chromatograph equipped with a thermal conductivity detector was used to measure the concentration of hydrogen gas (initially at 4.0%, certified) with a balance of nitrogen over a period of about 35 minutes.

Figure-6 shows a photograph of the test chamber where the hydrogen diffusion rate through the filter and membrane was measured. A precise mixture of hydrogen gas ( $\text{H}_2$ ) and nitrogen ( $\text{N}_2$ ) is circulated by a peristaltic pump through the gas chromatograph and returned to the test vessel. At the start of a test and at five minute intervals, a sample of the gas is injected into the detector for analysis. Seven samples are taken over a 35 minute period. The amount of hydrogen in the test vessel decays over time due to diffusion through the filter. The hydrogen diffusivity losses through tubing and connections have been measured and are about three orders of magnitude lower than diffusion rates through the filter. Therefore, no adjustments to filter diffusion coefficients are made.

The chromatogram peak area is related to the amount of hydrogen in the container. The decay rate, or hydrogen diffusivity,  $D$ , through the assembly is given by:

$$D = PV/t RT \ln(H_0/H_t)$$

Where  $P$  is atmospheric pressure,  $V$  is the volume of the vessel with connective tubing,  $t$  is elapsed time in seconds,  $R$  is the Ideal Gas Constant, and  $T$  is the temperature in Kelvin. The diffusion rate,  $D$ , is in moles/ second / mole fraction. The initial concentration of hydrogen given by the chromatogram peak area is  $H_0$  and the amount of hydrogen after time  $t$  is  $H_t$ .

A special test fixture was assembled that nearly duplicated the transport path of the gas through the filter and membrane in the containers. Chromatograms and spreadsheet calculations are shown in Appendix - 1 attached. Only two samples were measured for hydrogen diffusivity because the test is very long. The hydrogen transport rate through the filter and membrane averaged  $15.75 \text{ E-06}$  mole/ second/ mole

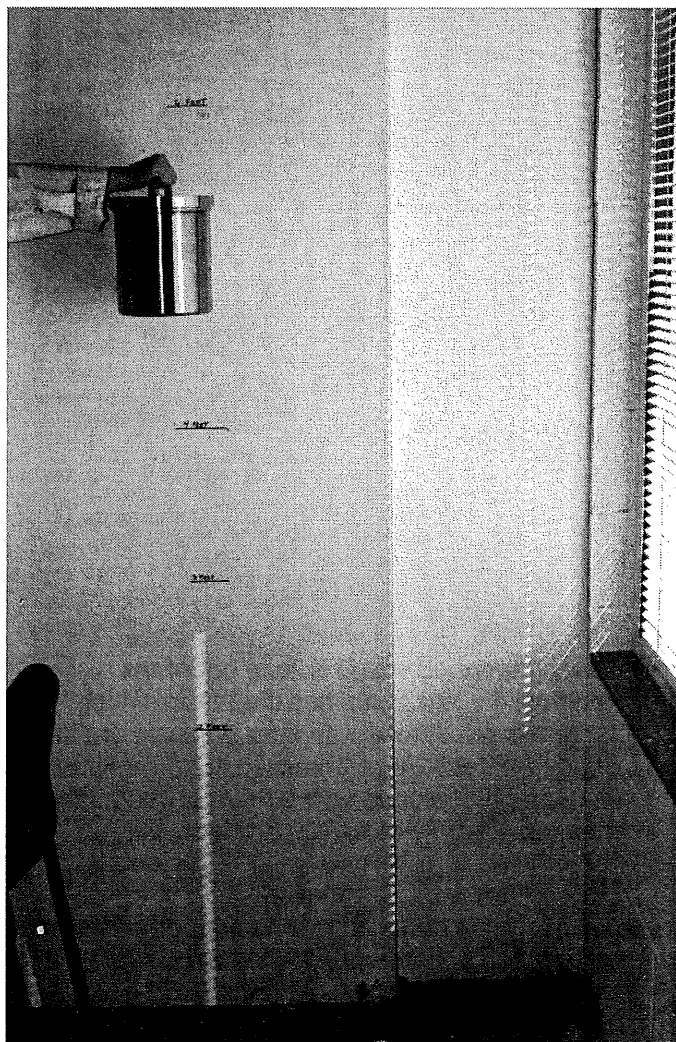


Figure -5 Drop Test From 1.21 Meters



Figure -6 Test chamber for hydrogen diffusion measurements

fraction which is 8 times greater than the minimum established by the WIPP TRUPACT II SAR. Oxygen peaks begin to appear after about 10 minutes which is the result of air diffusing back into the container.

#### **AIR PERMEABILITY AND FILTRATION EFFICIENCY**

Each container lid is equipped with a type 316 stainless steel HEPA grade filter media (a carbon-bonded-carbon filter may also be used). The lid, with the integral filter, is shown being tested for flow rate and particle removal efficiency in Figure - 7 and 8. Air permeability and filtration efficiency are measured using an Air Techniques Inc., aerosol photometer with a 0.3 to 0.5 micron dioctyl phthalate (DOP) aerosol. The optical photometer verifies that the filter vent qualifies as a HEPA grade filter, removal of greater than 99.97% of aerosol. Given an air flow rate of 200 ml/min across the filter, the resistance across the filter must be 1.0 inches water column or less. In production, all filters will be tested for air permeability and particle retention. Filter efficiency tests were conducted on 10 container lids with integral stainless steel filters. Listed in Table -2 are the results showing the airflow rates of 210 ml/min, percent penetration of DOP, and resistance to air flow (inches water column).

**TABLE -2**

<u>SERIAL I.D.</u>	<u>PERCENT PENETRATION</u>	<u>RESISTANCE TO FLOW (inches W.C.)</u>	<u>FLOW RATE ( ml/min )</u>
RFP-NMC-201	0.004	0.55	210
RFP-NMC-202	0.004	0.55	210
RFP-NMC-203	0.008	0.55	210
RFP-NMC-204	0.004	0.55	210
RFP-NMC-205	0.008	0.60	210
RFP-NMC-206	0.010	0.65	210
RFP-NMC-207	0.010	0.65	210
RFP-NMC-208	0.004	0.55	210
RFP-NMC-209	0.004	0.55	210
RFP-NMC-210	0.004	0.55	210

Of the first 1000 cans in production, there were no failures due to percent of penetration or high resistance to flow.

## **WATER ENTRY TEST**

To verify that no water intrusion will occur, for example from fire suppressant systems, the containers were subject to a 1 psig water column for a period of 12 hours. After a 12 hour period there was no visible water detected on the interior surface of the container. A second water intrusion test subjected the containers to a constant spray of water, at a rate of 30 gallons per minute for 1 hour, with no visible water entry. Figure -9 shows a photograph of the water spray test. A third water spray test utilized an industrial dishwasher where the canister was subjected to a high pressure spray (greater than 40 PSI) at all angle at approximately 90 degrees Celsius for 1.5 minutes. There was no visible water entry after the high pressure spray test. Figure -10 shows a photograph of the high pressure water spray generated from the industrial dishwasher.

## **CONCLUSION**

Nuclear materials storage containers fabricated entirely from type 304 stainless steel have been developed and tested to verify leak tightness under severe impact. The nuclear materials containers were designed to meet requirements established in DOE's Recommendation 94-1, "Criteria For Interim Safe Storage of Plutonium-Bearing Solid Material". Containers used for the interim storage (less than 20 years) of nuclear bearing materials must allow a means for retrieval and observation of stored contents, while also meeting stringent structural and leak-proof requirements. The new, all stainless steel, container has a threaded lid closure that is equipped with a sintered stainless steel HEPA filter vent. The hand tightenable container provides a positive seal for storing nuclear material, yet allows the controlled release of explosive gases including hydrogen.

The results of air-leak after a 1.21 meter drop test indicates that the container seal was not compromised from the drop impact. Hydrogen diffusivity measurements indicate hydrogen gas will be transported through the integral filter and semipermeable membrane at a rate of about  $15 \text{ E-06}$  moles/second/mole fraction. Filter efficiency tests demonstrate that a reliable seal is formed with the sintered stainless steel filter media. Particle retention of 0.3 to 0.5 micron DOP aerosol was measured at greater than 99.97% at an air flow of greater than 210 milliliters per minute. Through three different water entry test, one pressurized at 1 PSIG and two water spray tests, it is demonstrated that water will not enter the container.

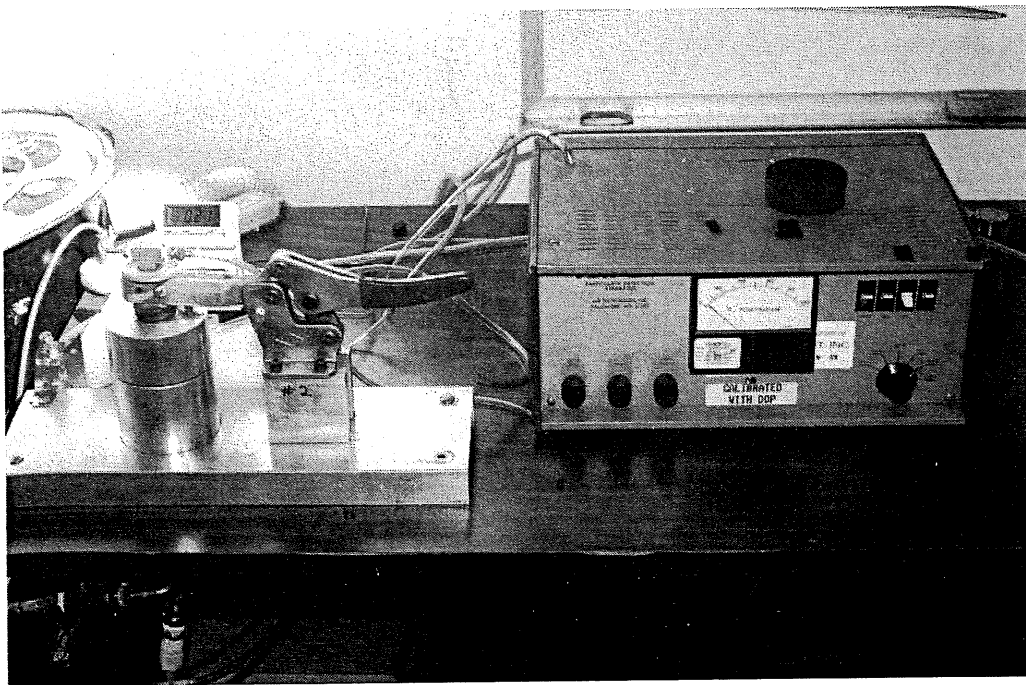


Figure -7 Optical photometer used for filter Efficiency test

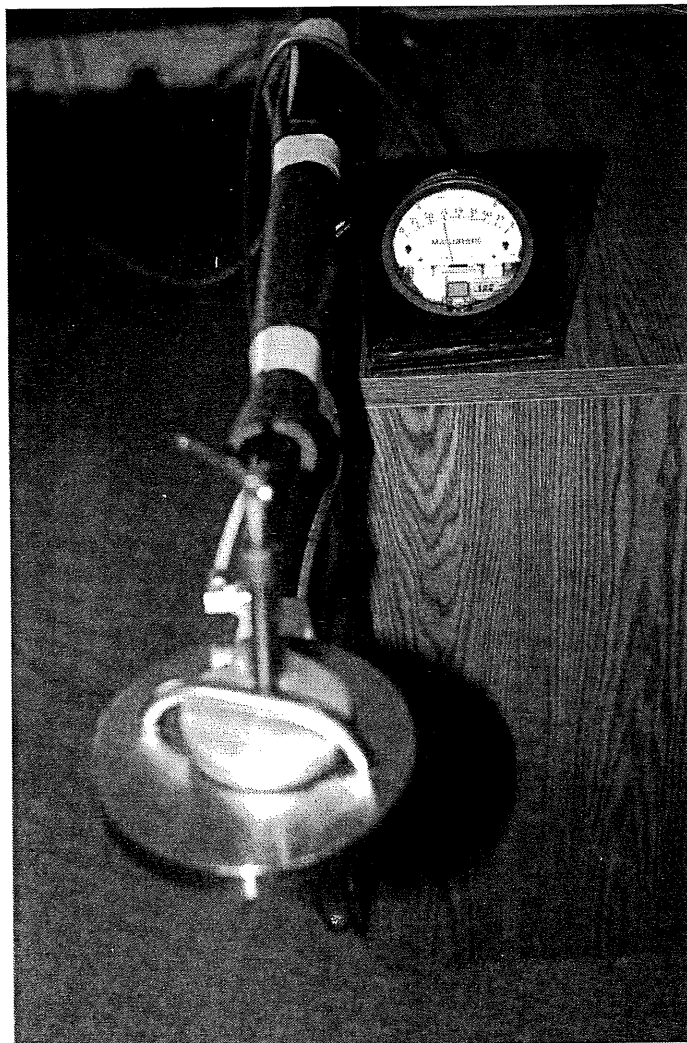


Figure 8 Cassette lid with internal filter system

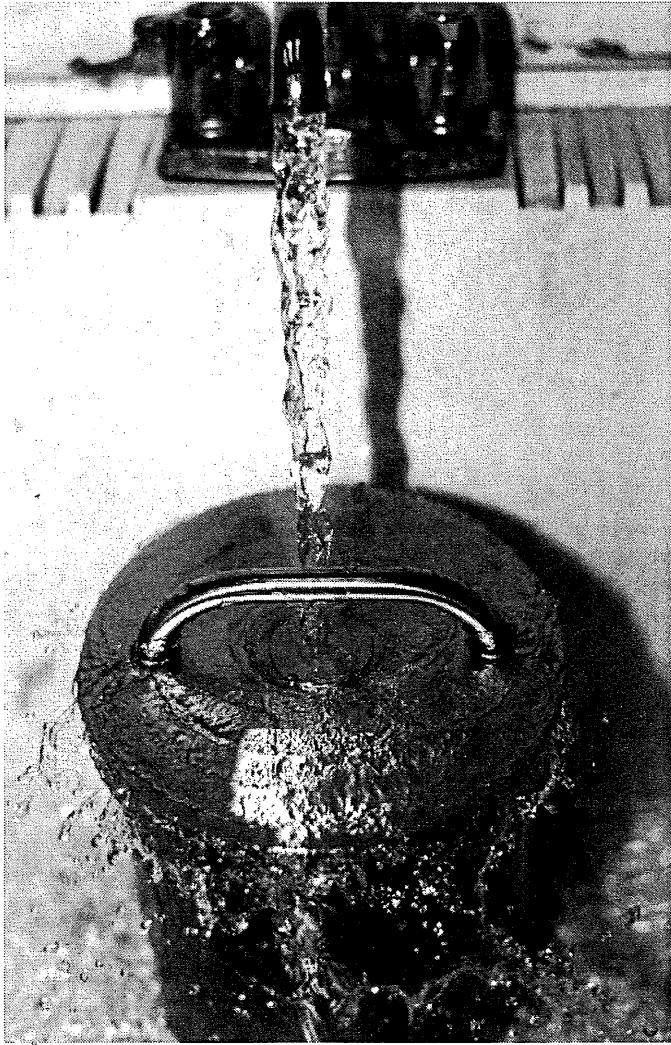


Figure -9 NMC under water spray test

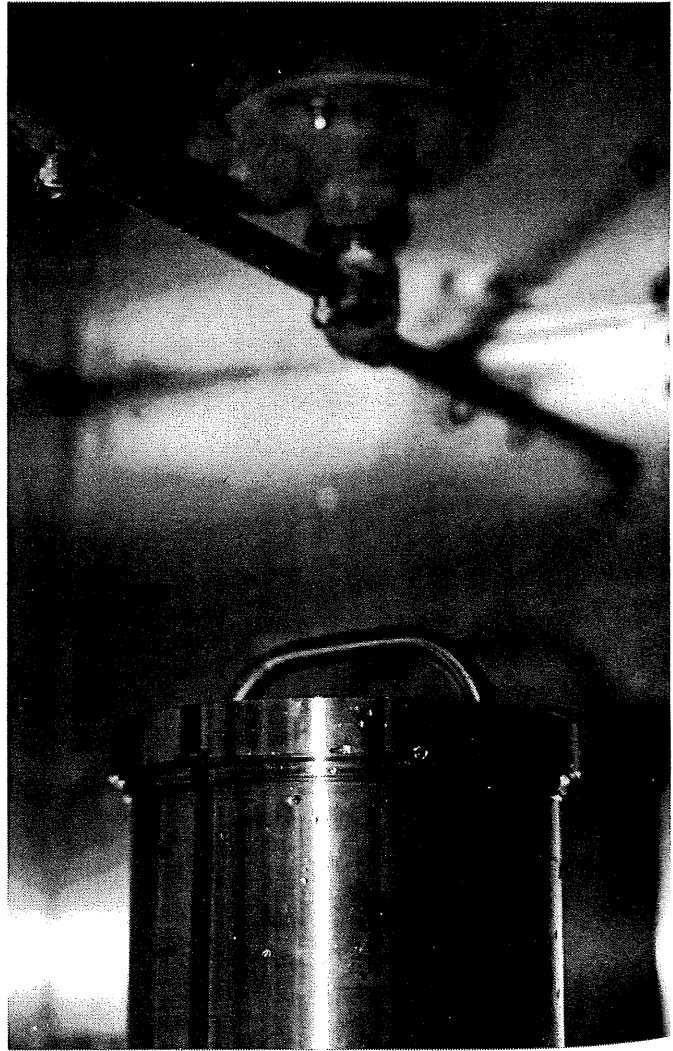


Figure -10 NMC in high pressure industrial water spray test

## APPENDIX -1

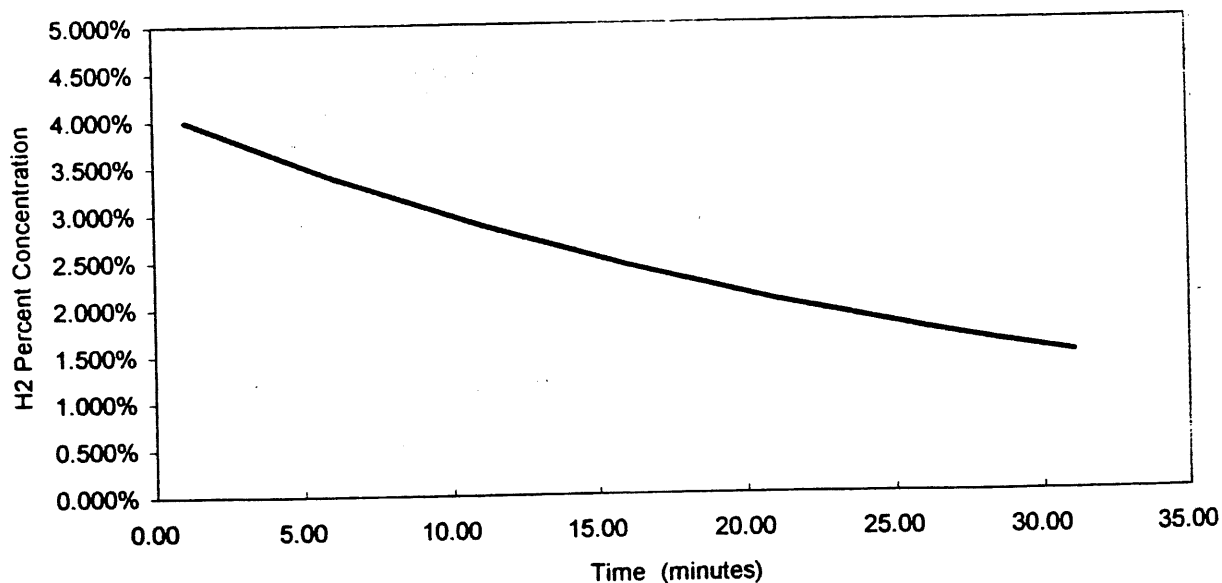
## Hydrogen Diffusion Chromatograms and Spreadsheet Calculations

DATE: 08-Apr-96 FORM: DIFFCALC.WQ1  
 USER: Terry W.  
 FILE: C:\qpro\diff\sss200  
 SAMPLE I.D Sss 200 4/9/! MODEL: NUCFIL-013  
 ATM. P (atr 1  
 TEMP. (K) 297.6  
 GAS CONSTAI 0.08206  
 VESS. VOL 0.813

RESULTS	
I.D.:	Sss 200 4/9/96
Diff Constant:	1.495E-05
Min Accepted:	1.900E-06
ACCEPT/REJECT	ACCEPT

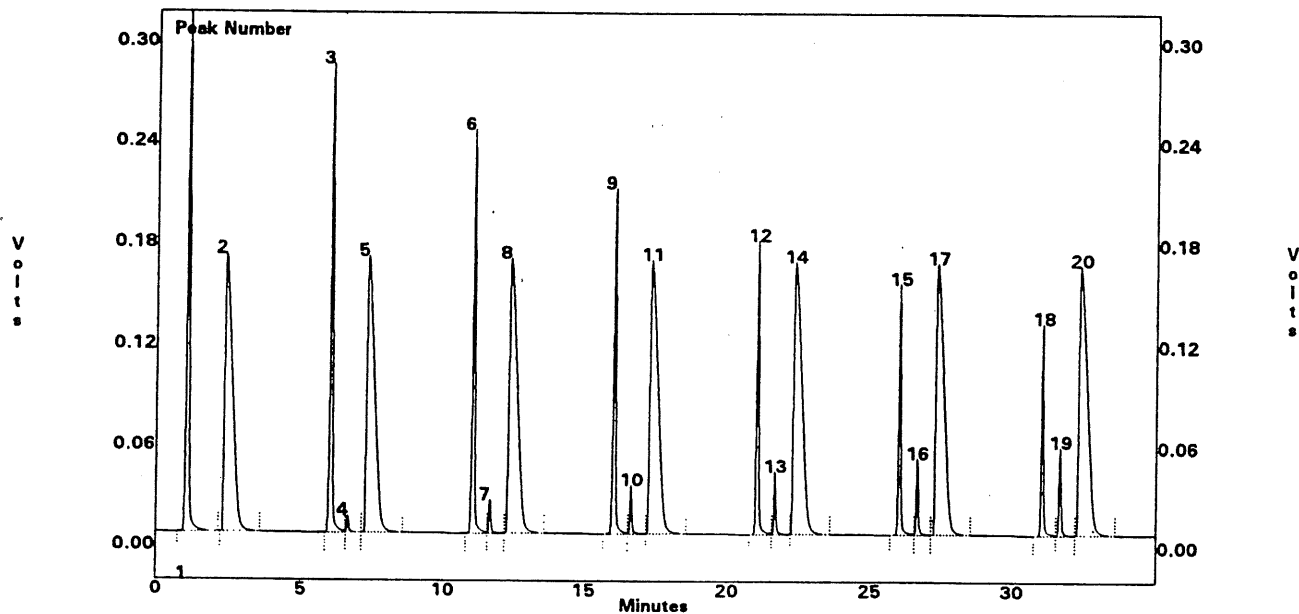
H2 Pk	Ret. Time	Time sec)	AREA	Percent	DATE
-	-	-	-	-	Diffusion Constant Mol/MolFrac/s
H2 Initial	1.07	0.00	1780125	3.990%	#DIV/0!
H2 @t=5min	6.05	298.80	1510618	3.386%	1.483E-05
H2@t=10min	11.05	598.80	1280486	2.870%	1.486E-05
H2@t=15min	16.05	898.80	1084351	2.430%	1.490E-05
H2@t=20min	21.05	1198.80	914869	2.051%	1.503E-05
H2@t=25min	26.05	1498.80	774929	1.737%	1.501E-05
H2@t=30min	31.05	1798.80	652840	1.463%	1.510E-05
Average....					1.495E-05
STDV.....					9.922E-08
Range.....					2.743E-07

Hydrogen Diffusion Rate Through NucFil  
 % Hydrogen Concentration Vs. Time



Method : C:\EZCHROM\chrom\diffsh.MET  
Sample ID : STRD-2 NUCFIL-013 SINTERED 20  
Acquired : Apr 08, 1996 14:28:42  
Printed : Apr 08, 1996 15:04:03  
User : Terry W.

C:\EZCHROM\CHROM\SSS200 -- Channel A



Channel A Results

Peak	Name	Time	Area
1	Hydrogen Initial	1.08	1780125
--	Oxygen Initial	1.65	0
2	Nitrogen Initial	2.34	3241179
3	H2 @ t = 5 minutes	6.07	1510618
4	O2 @ t = 5 Minutes	6.67	54667
5	N2 @ t = 5 Minutes	7.34	3208727
6	H2 @ t = 10 Minutes	11.06	1280486
7	O2 @ t = 10 Minutes	11.67	101196
8	N2 @ t = 10 Minutes	12.34	3177777
9	H2 @ t = 15 Minutes	16.06	1084351
10	O2 @ t = 15 Minutes	16.67	147762
11	N2 @ t = 15 Minutes	17.35	3151418
12	H2 @ t = 20 Minutes	21.06	914869
13	O2 @ t = 20 Minutes	21.67	188264
14	N2 @ t = 20 Minutes	22.35	3113861
15	H2 @ t = 25 Minutes	26.06	774929
16	O2 @ t = 25 Minutes	26.67	226979
17	N2 @ t = 25 Minutes	27.35	3091258
18	H2 @ t = 30 Minutes	31.06	652840
19	O2 @ t = 30 Minutes	31.68	264343
20	N2 @ t = 30 Minutes	32.36	3057786

Totals :

31023446

DATE: 08-Apr-96 FORM: DIFFCALC.WQ1  
 USER: Terry W.  
 FILE: C:\qpro\diff'#54sss  
 SAMPLE I.D SINTST.ST.01: MODEL: NUCFIL-013  
 ATM. P (atm) 1  
 TEMP. (K) 297.6  
 GAS CONSTAI 0.08206  
 VESS. VOL 0.813

QA

DATE

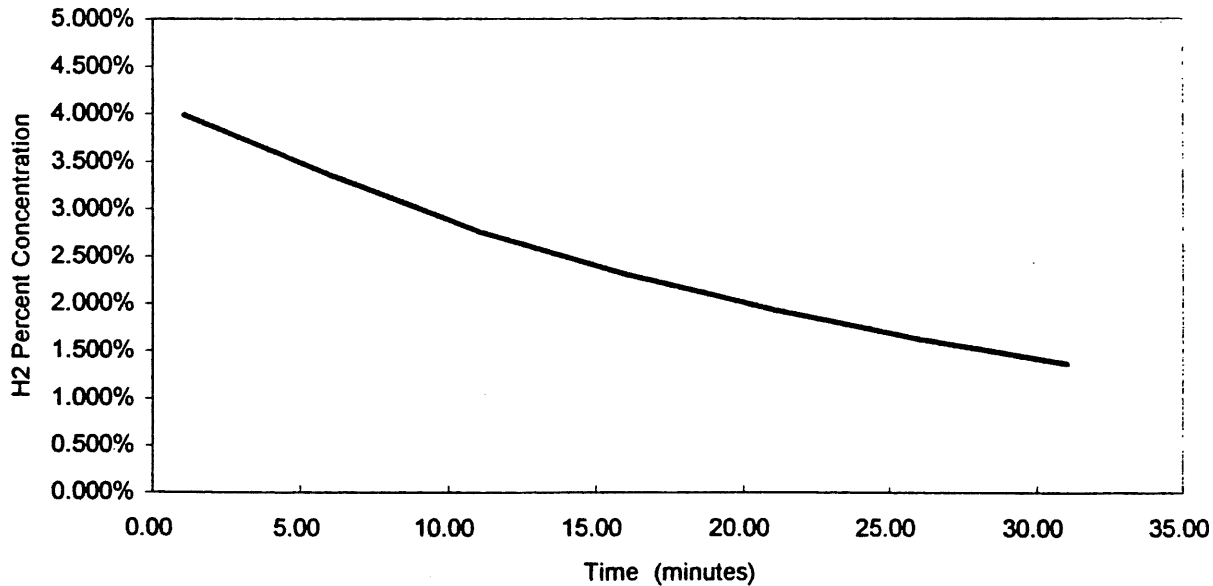
## RESULTS

I.D.:	SINTST.ST.013SS
Diff Constant:	1.656E-05
Min Accepted:	1.900E-06
ACCEPT/REJECT	ACCEPT

Diffusion  
Constant

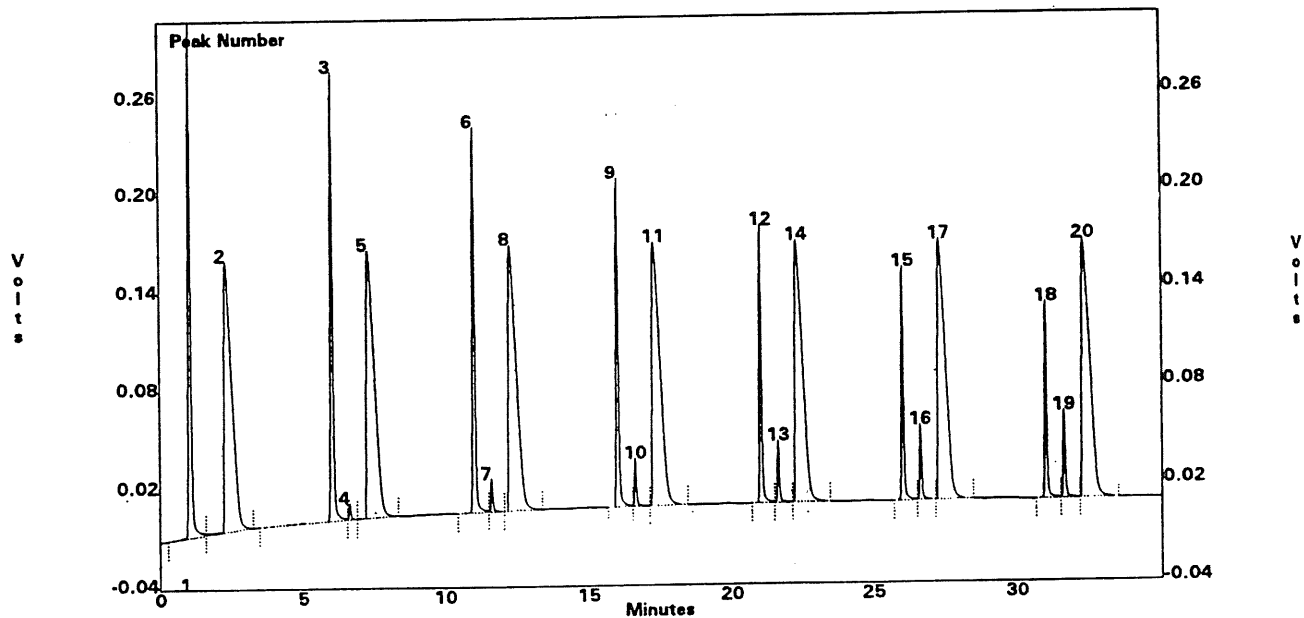
H2 Pk	Ret. Time	Time sec)	AREA	Percent	Mol/MolFrac/S
H2 Initia	1.07	0.00	1807004	3.990%	#DIV/0!
H2 @t=5min	6.05	298.80	1519056	3.354%	1.588E-05
H2@t=10min	11.05	598.80	1250552	2.761%	1.700E-05
H2@t=15min	16.05	898.80	1045646	2.309%	1.680E-05
H2@t=20min	21.05	1198.80	875293	1.933%	1.667E-05
H2@t=25min	26.05	1498.80	734648	1.622%	1.653E-05
H2@t=30min	31.05	1798.80	615775	1.360%	1.646E-05
Average....					1.656E-05
STDV.....					3.514E-07
Range.....					1.124E-06

Hydrogen Diffussion Rate Through NucFil  
 % Hydrogen Concentration Vs. Time



Method : C:\EZCHROM\chrom\diffsn.MET  
Sample ID : Sintered SS #54 2nd run 4/9/9  
Acquired : Apr 08, 1996 06:28:21  
Printed : Apr 08, 1996 07:03:48  
User : Terry W.

C:\EZCHROM\CHROM\#54ss2 - Channel A



# Channel A Results

Peak	Name	Time	Area
1	Hydrogen Initial	1.07	1807004
--	Oxygen Initial	1.65	0
2	Nitrogen Initial	2.32	3266442
3	H2 @ t = 5 minutes	6.05	1519056
4	O2 @ t = 5 Minutes	6.65	56765
5	N2 @ t = 5 Minutes	7.31	3177959
6	H2 @ t = 10 Minutes	11.05	1250552
7	O2 @ t = 10 Minutes	11.65	104877
8	N2 @ t = 10 Minutes	12.31	3130961
9	H2 @ t = 15 Minutes	16.05	1045646
10	O2 @ t = 15 Minutes	16.65	148489
11	N2 @ t = 15 Minutes	17.31	3091453
12	H2 @ t = 20 Minutes	21.05	875293
13	O2 @ t = 20 Minutes	21.65	190839
14	N2 @ t = 20 Minutes	22.32	3054571
15	H2 @ t = 25 Minutes	26.05	734648
16	O2 @ t = 25 Minutes	26.65	229171
17	N2 @ t = 25 Minutes	27.32	3023443
18	H2 @ t = 30 Minutes	31.04	615775
19	O2 @ t = 30 Minutes	31.65	265684
20	N2 @ t = 30 Minutes	32.33	2991993

Totals :

30580634